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AMERICAN DYESTUFF REPORTER

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of the
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VOLUME 29 JULY 22, 1940 NUMBER 15

The Relation of Cation Exchange to the Acidic Properties of Cotton

ARNOLD M. SOOKNE and MILTON HARRIS*

ABSTRACT

It has long been known that cellulosic materials exhibit acidic properties which influence many of their physical and chemical characteristics. Thus, the acidic groups of the fiber bind small amounts of ash which have a considerable effect on the electrical conductivity of cotton fibers, and on the viscosities of solutions of some cellulose derivatives. The ash may also affect the dyeing properties of the fiber, and may be related to the application of finishing agents. Since the ash of washed cotton consists principally of cations held by acidic groups, the problems of measuring the ash and the number of acidic groups on the fiber are intimately related. In addition, if some of the acidic groups of purified cotton are a part of the cellulose molecule, such as end-groups, the number of these groups may be a measure of its chain length, which is of primary importance in determining the strength and other physical characteristics of the fiber.

As part of a general program for the investigation of the acidic and basic properties of textile fibers, a study has been made of the manner in which hydrochloric acid is bound by cotton, and of the dependence of the amount of acid bound on the concentration of the acid solution in contact with the fiber. The effect on this dependence of different amounts of an added neutral salt, potassium chloride, has also been studied.

A comparison of the acid bound with the cationic ash (the total content of the cations of inorganic bases) as determined by an electrodialysis method leads to the conclusion that the maximum acid-binding capacity is equivalent to the cationic ash, and that therefore the acid bound at any acidity depends upon the ash. A comparison of the results for dewaxed cotton with those for depectinized cotton shows that most of the acidic groups of cotton are contributed by the pectic substance.

Cotton freed of pectic substance retains a small number of acidic groups. Although other possibilities exist, it seems likely that these are an integral part of the cellulose molecule, such as an end-group. If this is actually the case, the results show that the cotton cellulose molecule has an equivalent weight of about 100,000, which corresponds to a minimum chain length of about 600 glucose residues.

I. INTRODUCTION

COTTON and other naturally-occurring cellulosic materials yield, on ignition, an ash which is alkaline in reaction. This results from the fact that the fiber normally exists as a salt, many of the anions being provided by the cellulose of the fiber and by associated materials such as pectic substance[1, 2, 3]. The cations in water-washed cotton have been shown by Walker and Quell to consist principally of Ca^{++}, Mg^{++}, Fe^{+++}, and Al^{+++}[2]. In addition, raw cotton also contains a quantity of neutral salt, most of which is removed during washing of the fiber with water.

The manner in which cotton binds acid may be considered as an exchange process between the hydrogen ions of the acid and the cations associated with the fiber. The extent of the exchange is a function of the number and nature of the cations, the concentration of the acid, and the tendency of the hydrogen of the acidic groups in the cotton to ionize. This concept of cation exchange has been employed from time to time by earlier investigators to explain the reaction of cellulosic materials with acids and salts[1,2,3,4]. The importance and wide applicability of these exchange reactions have been recognized and discussed by McLean and Wooten[3] who demonstrated that the amount of acid bound by different cellulosic materials at an arbitrary pH is proportional to their ash contents. They also suggested

*Research Associates at the National Bureau of Standards, representing the Textile Foundation, Inc.

that the major portion of the ash is contributed by non-cellulosic materials associated with the cellulose.

In the present investigation, which involved a detailed study of the acid-binding capacity of cotton fibers over a wide range of pH values, it is shown that the acid-binding capacity of cotton at any pH is determined by its cationic ash. From the complete titration curves, and the hydrogen ion equivalence of the ash, the maximum acid bound by different samples of cotton was ascertained, which in turn permitted the quantitative allocation of the acid bound to the principal constituents of the fiber.

II. EXPERIMENTAL PROCEDURE

1. Materials

Raw cotton was extracted with hot alcohol for 24 hours, and then washed thoroughly with distilled water at room temperature for several hours. Such samples will hence-forth be described as *dewaxed cotton.*

A portion of the dewaxed material was further purified by extraction for 4 or 8 hours with a boiling 1 per cent solution of sodium hydroxide, according to the method recommended for the preparation of standard cellulose. The procedure was essentially the same as that described by Corey and Gray[5] except that the apparatus of Worner and Mease[6] was used. Fibers treated in this way were shown by the method of Whistler, Martin, and Harris[7] to be free of pectic substance. In one step of this purification, the fibers were washed with a dilute solution of acetic acid. This undoubtedly removed some of the cations from the fiber and since cotton binds acid by a cation-exchange process which involves the back titration of the acid groups of the fiber by an added acid, it was necessary to make certain that all of the acidic groups were in the salt form. This was accomplished by soaking the purified samples for about 3 hours in a solution of calcium hydroxide at room temperature. They were then washed in running distilled water for about 24 hours. Cotton from which both wax and pectic substance were removed is designated *depectinized cotton.* Depectinized cotton which has been washed with a solution of calcium hydroxide is designated *lime-washed depectinized cotton.*

All samples were conditioned at 21° C. and 65 per cent relative humidity before weighing. The moisture contents of the fibers were determined by drying representative samples at 105° C. for 2 hours in a vacuum oven.

2. Methods

(a) *Determination of the Acid-binding Capacity.* Samples of cotton weighing about 5 g. and containing a known amount of moisture were immersed in 100-ml. portions of the acid solutions at the temperature of the experiment (0° or 25° C.). Large test-tubes (1½" x 8") were found to be convenient for this purpose, since the samples could be easily pressed into the bottom of the tube to insure uniform wetting of the fibers or to permit squeezing the liquor from the fibers after equilibrium was reached. The amount of acid absorbed was determined by titration with sodium hydroxide of 50-ml. aliquots of the original solution and of the solution in equilibrium with the sample, using bromcresol purple as the indicator. From the difference between these titers, the weight of the sample, and its content of moisture, the amount of acid taken up per gram of dry cotton was calculated. In a number of measurements, the concentrations of strongly acid solutions (in the region of pH 1) were determined by adding a weighed quantity of strong alkali to the solution, after which the titration was completed with weak acid or alkali.

Preliminary measurements at several acidities indicated that a close approximation to a final equilibrium is attained in a few hours. In most of the experiments, however, the samples were allowed to remain in contact with the solutions overnight.

(b) *Determination of pH.* Measurements of pH were made with a McInnes and Belcher-type glass electrode and a vacuum-tube potentiometer, using a cathode-ray "eye" as null indicator. The pH values were referred to potassium acid phthalate, 0.05 *M*, to which was assigned a pH value of 4.01[8].

(c) *Temperature.* With the exception of one experiment at 25° ± .02° C., all of the acid solutions in contact with the cotton were kept at 0° ± .01° C. to inhibit decomposition of the fibers.

(d) *Determination of Cationic Ash.* The hydrogen ion equivalence of the cationic ash was determined by an electrodialysis method described elsewhere[9].

III. RESULTS AND DISCUSSION

1. Selective Sorption of Water by Cotton

The determination of the maximum acid-binding capacity of cellulose is complicated by the fact that the fibers may selectively sorb water from a solution of acid, and thus affect its titer. Since the acid-binding capacity is calculated from titer differences, a correction must be applied to compensate for this effect. At pH values below 1.5, the magnitude of the correction is appreciable compared with the acid-binding capacity of cotton, but becomes negligible at pH values above 2.5.

If B is the amount of water selectively sorbed per gram of dry cotton, then the amount sorbed per gram from the solution by a sample which originally contains W g. of water per gram is (B-W). Water taken up in this manner is no longer available for dilution of the acid, and would thus tend to cause an increase in concentration of the acid solution in contact with the fiber. At the same time, however, a quantity of acid A (milliequivalents per gram), will be bound by the sample, and produce a decrease in concentration of the acid solution. Obviously, if any of the acid solution as such is dissolved in the sample, the titer of the solution will not be affected. From the above, it follows that if S g. of dry cotton are immersed in G ml. of acid solution of concentration C_0, and the concentration of the solution after equilibrium is attained is C, the corrected acid-binding capacity of the cotton is given by the equation,

$$A = \frac{G\,(C_0 - C)}{S} + C\,(B\text{-}W) \qquad (1)$$

where A is the corrected acid-binding capacity, $\frac{G(C_0 - C)}{S}$ is the apparent acid-binding capacity, and C(B-W) is the correction for the selective sorption of water.

The actual measurement of the quantity B presents considerable experimental difficulty. In the region of the maximum acid-binding capacity, measurement of titer differences in solutions of different initial acid concentrations, or in solutions of the same initial acid concentration which have been allowed to come to equilibrium with samples of different moisture contents, gives equations of the form of equation (1). The solution of any two of such equations should give values for A and B, but such a procedure demands the assumption that both A and B are constant for the conditions employed. Measurements made in 0.1 and 0.2 *M* solutions of hydrochloric acid indicated that this assumption is not valid. The results were such as to indicate that more acid or less water was sorbed in the more concentrated of these solutions. If calculations are made from measurements of solutions for which the above assumption should be more nearly valid (0.10 and 0.11 *M* solutions of hydrochloric acid) the values for A and B are too sensitive to experimental error to be useful.

It was found, however, that the following method for evaluating A and B leads to less equivocal results. A substance which is assumed not to be sorbed by the fiber was added to the solution of acid as a reference substance. The change in concentration of this substance gives a direct measure of the amount of water (B-W) sorbed by the fiber. Two reference substances, sodium chloride and trehalose, were used in this work. The change in concentration of the former was determined by evaporating an aliquot of the solution and determining the dry weight of salt. In the case of the disaccharide, trehalose, the change in concentration was measured polarimetrically. The actual determinations were made as follows. Samples of cotton of known weights and moisture contents were immersed in solutions that were 0.1 *M* with respect to hydrochloric acid, and 3.43 *M* with respect to sodium chloride, and the changes in concentration of both acid and salt were determined. The acid-binding capacity, A, was then calculated, due account being taken of the water sorbed as determined by the change in salt concentration. The mean of four values, so determined, was 0.067 milliequivalent per gram.

The value for B in solutions in the region of pH 1 was then calculated by substituting the value 0.067 for A in equation 1. The value $(C_0 - C)$ is small, and therefore the determination of B by this procedure is subject to the uncertainty of this quantity. In order to improve the reliability of B, a series of 20 samples of dewaxed cotton of known weights and moisture contents was immersed in solutions of hydrochloric acid of known concentrations, the acidities of which were in the region of pH 1. The 20 equations of the form of equation (1), obtained from these measurements, were summed up and gave the equation,

$$28.63A - 3.87B = 1.489 \qquad (2)$$

Substitution of the value of 0.067 for A in equation (2) gave a value for B (in HCl solution) of 0.11 g. of water per gram of dry cotton.

The values of A and B were also determined by a second method, in which trehalose was used as the reference substance. Samples of cotton of known weights and moisture contents were immersed in trehalose solutions that were 0.04 *M* with respect to hydrochloric acid. The concentration of trehalose before and after the introduction of the samples was measured polarimetrically. By this procedure, a value for B of 0.13 g. of water per gram of dry cotton was obtained.

The validity of this method of correction for the selective sorption of water is shown in Table 1, which presents data on dewaxed fibers of widely different initial moisture contents. The measurements were made in 0.1 *M* solutions of hydrochloric acid, and the results are corrected using the mean value, 0.12 for B. Each figure represents the mean of at least two determinations. In the cases of the samples of high moisture contents, some of the water was added directly to the solution with the sample. The discordant values for the apparent acid-binding capacity, $\frac{G(C_0 - C)}{S}$ for all of these samples are brought into close agreement by application of the correction C(B-W), to give the corrected acid-binding capacity, A.

TABLE 1

Application of the Correction for the Selective Sorption of Water to the Acid-binding Capacity of Dewaxed Cotton in 0.1 *M* Hydrochloric Acid

Initial Water Content	*Apparent Acid-Binding Capacity* $\frac{G(C_0 - C)}{S}$	*Correction* $C(B - W)$	*Corrected Acid-binding Capacity* A
g./g. Cotton	*Milliequiv./g.*	*Milliequiv./g.*	*Milliequiv./g.*
0.000	0.054	+0.012	0.066
.100	.063	+ .002	.065
.190	.075	— .007	.068
.308	.085	— .019	.066
.720	.124	— .059	.065
1.135	.164	— .097	.067
			0.066 Mean

It is of interest to note that the values for B, 0.11 and 0.13 g. of water per gram of dry cotton, are considerably lower than the known moisture content of cotton at relative humidities between 99 and 100 per cent (about 0.3 g. of water per gram of cotton)[10], which corresponds approximately to the humidity of air in equilibrium with 0.1 *M* hydrochloric acid. The result suggests that the average partition coefficient of the hydrochloric acid between the water in the external solution and the water held by the fiber is greater than unity. The average value of B of 0.12 g. of water per gram of cotton represents the portion of the total sorbed water which is unavailable for dilution of the acid.

2. Combination of Dewaxed and of Lime-washed, Depectinized Cotton with Hydrochloric Acid

Measurements of the combination of dewaxed and of lime-washed, depectinized cotton with hydrochloric acid are given in Table 2 and shown graphically in Figure 1.

TABLE 2

Combination of Dewaxed and of Lime-washed, Depectinized Cotton with Hydrochloric Acid at 0° C., in the Presence of Varying Amounts of Potassium Chloride

Dewaxed Cotton

No Added Salt		*0.1 M Ionic Strength*		*1.0 M Ionic Strength*	
pH	*Acid Bound*	*pH*	*Acid Bound*	*pH*	*Acid Bound*
	Milliequiv./g.		*Milliequiv./g.*		*Milliequiv./g.*
1.08	0.066	1.08	0.066	2.03	0.056
1.47	.061	1.48	.062	2.55	.046
1.96	.058	1.98	.057	3.06	.036
2.47	.052	2.45	.048	3.56	.024
3.05	.041	2.94	.040	4.05	.012
3.64	.025	3.39	.030	4.62	.005
4.45	.011	3.70	.018	5.24	.001
5.22	.004	4.02	.011	5.72	.000
5.78	.001	4.44	.006		
6.04	.000	4.52	.004		
6.30	.000	4.69	.003		
		5.20	—.001		

Lime-washed Depectinized (Four Hour) Cotton

1.08	0.008	1.08	0.008	2.04	0.006
1.51	.007	1.53	.005	3.00	.004
2.04	.006	2.36	.003	4.03	.001
2.52	.005	3.15	.002	5.18	.000
3.00	.004	3.90	.001	5.84	.000
3.48	.004	4.16	.001		
4.09	.003	4.63	.000		
4.71	.002	5.52	.000		
5.67	.000				
6.27	.000				
6.38	.000				

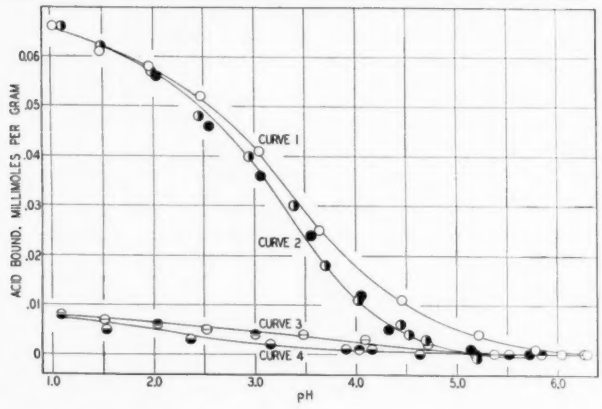

Figure 1. Combination of dewaxed and of lime-washed depectinize cotton with hydrochloric acid at 0° C. Curve 1, dewaxed cotton in the absence of added salt, ○. Curve 2, dewaxed cotton in hydrochloric acid-potassium chloride solutions of 0.1 *M* ionic strength, ◑; and in solutions of 1.0 *M* ionic strength, ●. Curve 3, depectinized cotton in the absence of added salt, ⊖. Curve 4, depectinized cotton in solutions of 0.1 *M* ionic strength, ◒; and in solutions of 1.0 *M* ionic strength, ⊖.

The data were obtained in the absence of added salt, and in solutions made up to 0.1 *M* and 1.0 *M* ionic strengths with potassium chloride. The increase of ionic strength from 0.1 to 1.0 *M* produced no measurable effect on the titration curve of either sample, and accordingly only one curve is drawn for each sample at the two ionic strengths. The curves for dewaxed and for lime-washed, depectinized cotton tended to approach maxima, at about pH 1, of 0.066 and 0.008 milliequivalent per gram, respectively. The failure to arrive at unambiguous maxima may be due to the uncertainty in the correction for the selective sorption of water or to other secondary effects which may occur in solutions of high acid concentrations.

None of the pectic substance appeared to dissolve during the titration with acid. This was indicated by sharp indicator endpoints, lack of frothing, exact correspondence between acid-binding capacity calculated from pH differences and from titer differences, and the pH of 0.1 *M* potassium chloride solution in contact with the sample (about 5.3). If any soluble potassium pectate had been formed, the pH value of the solution would necessarily have been appreciably higher. The pectic substance may be insoluble because it exists as an insoluble salt of polyvalent cations or as the free acid, which is insoluble in acid solutions.

The difference between the maxima of the curves indicates that about 85 per cent of the acid groups of dewaxed cotton is furnished by the pectic substance. This conclusion is confirmed by direct chemical analyses presented elsewhere. The remaining acid groups appear to be associated with the depectinized cotton. Further consideration of these groups and their probable significance is given later.

The titration curves for dewaxed cotton (curves 1 and 2, Figure 1) must be considered as composites of the curves for depectinized cotton and the pectic substance. Accordingly, subtraction, at corresponding values of pH, of the ordinates of curve 3 from those of curve 1 should give a titration curve for the pectic substance in the absence of added salt. Similarly, subtraction of the ordinates of curve 4 from those of curve 3 gives the curve for pectic substance in the presence of added salt (0.1 or 1.0 *M* potassium chloride). The data thus obtained interpolated at intervals of 0.5 pH unit, are shown by the points in Figure 2. The

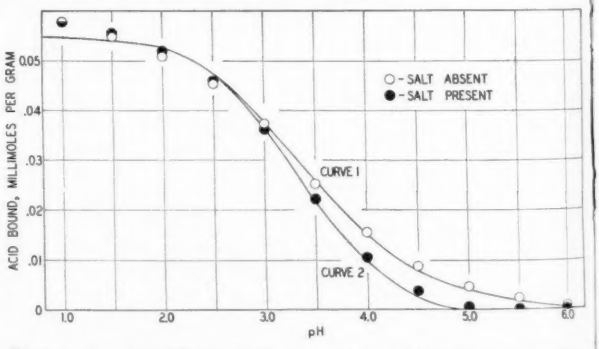

Figure 2. Calculated data for the combination of pectic substance with hydrochloric acid at 0° C. Curve 1, pectic substance in the absence of added salt, ○. Curve 2, pectic substance in solutions of 0.1 *M* or 1.0 *M* ionic strength, ●. The smooth curves are not drawn to fit the data, but are explained in the text (Section III—6).

(Continued on page 383)

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Annual Meeting and Convention, auspices New York Section, October 18th and 19th, 1940, Hotel Commodore, New York City

Meeting, Piedmont Section, Carolina Hotel, Pinehurst, N. C., November 1 and 2, 1940.

AATCC

LAPEL BUTTONS

These buttons, bearing the seal of the Association, can be purchased by members in good standing from the Secretary at $1.50 each.

MEETING, NORTHERN NEW ENGLAND SECTION

A MEETING of the Northern New England Section was held at the Andover Country Club, Andover, Massachusetts on Friday, May 10, 1940. There were fifty-one members present at the dinner and approximately sixty members attended the business meeting and technical program which followed.

Howard L. Jenkins of Standard Brands, Inc., New York, presented a paper entitled, "Application of Enzymes in the Textile Industry." At the close of Mr. Jenkins' talk an interesting discussion took place regarding various problems which have arisen with the use of enzymes in the textile industry. Mr. Jenkins very kindly responded to many of the questions of the members.

Respectfully submitted,

JOHN N. DALTON, *Secretary.*

MEETING, SOUTH CENTRAL SECTION

THE Spring Meeting of the South Central Section was held at the Farragut Hotel in Knoxville, Tenn., on Saturday, May 25, 1940. The principal speaker was Andrew J. Kelly of the Burkart-Schier Chemical Co., on, "Surface Active Agents in Wet Processing." Motion pictures from "Field & Stream" were shown. Committees were appointed and plans discussed for the Annual Summer Outing, to be held at the Lookout Mountain Hotel on August 2 and 3, 1940.

Respectfully submitted,

FRANK F. MYERS, *Secretary.*

REPORT, LOWELL TEXTILE INSTITUTE STUDENT CHAPTER

SINCE the last report, this chapter has held three meetings.

On April 8, 1940, William G. Chace of the Institute faculty addressed the members on the subject of enzymes.

On April 22, 1940, Willard A. Colby of Danvers Bleachery spoke on the bleaching, dyeing, and finishing of fabrics.

Election of officers for the year 1940-41 was held May 16, 1940. George McTeague was elected chairman and S. S. Puliafico was elected secretary. The new chairman appointed a program committee consisting of George Turner, chairman; Saunder Finard, and Ernest James.

Respectfully submitted,

NEIL MANNING, *Secretary.*

MEMBERSHIP APPLICATIONS

Senior

Stanley N. Galliano—Dyer, Arcadia Knitting Mills, Allentown, Pa. Sponsors: A. E. Raimo, A. W. Etchells.

Frank R. Hutt—Supt. of Dyeing, Woolen Div., Collins & Aikman Corp., Philadelphia. Pa. Sponsors: C. H. Knapp, Jr., C. T. Wagner, Jr.

Howard J. Rupright—Supt. of Production of Vat Dyes, Dow Chemical Co., Midland, Mich. Sponsors: A. F. Lichtenstein, H. B. Marshall.

Morton Schwartzman—Manager, Great American Color Co., Los Angeles, Cal. Sponsors: D. F. Driscoll, D. C. Metcalf.

Junior

Leon Albion Stanley, Jr.—Overseer of Dyeing, Bleaching & Polishing, The Linen Thread Co., Blue Mountain, Ala. Sponsors: R. N. Mallard, W. F. Crayton.

Waiter B. Worsman—Dyer, Wood Worsted Mills, American Woolen Co., Lawrence, Mass. Sponsor: S. E. Shepard, F. A. Dushame.

Associate

George A. Jonic—Colorist, Calco Chemical Div., American Cyanamid Co., Chicago, Ill.

ANNUAL OUTING, MID-WEST SECTION

THE tenth annual outing of the Midwest Section was held at Lake Lawn, Delavan, Wisconsin, June 22nd, 1940. There were 106 members and guests present.

The outing committee, under the capable leadership of James Morrison, did a fine job of handling the outing. They received a rising vote of thanks.

Leading scores in the golf tournament are as shown:

Millmen's Cup—Low Net Blind Handicap
Gay Brinn (winner by toss over) 101-36-65
Harold Tucker 96-31-65

Millmen's Trophy—Low Gross
Kenneth Harmes 81

Low Gross Trophy—Donated by Atlas Electric Devices Co.
Elmer Smith 78

Atlas Electric Devices Trophy to be donated for low gross score of members. Trophy must be won three years for permanent possession.

There were 62 prizes awarded for golf, and the donors of these were sent letters of appreciation.

Respectfully submitted,

DAVID A. ANDERSON, *Secretary.*

ANNUAL MEETING AND CONVENTION

OCTOBER 18, 19, AND 20

Atmospheric (Gas) Fading of Colored Cellulose Acetate*

CHARLES A. SEIBERT**

PART I

SUFFICIENT evidence has accumulated to establish as an acceptable fact, that the color destruction, which is the subject of this discussion, is caused by the presence in the atmosphere of oxides of nitrogen. As confirmatory evidence, if that is required or necessary, is the well known fact that the color changes can be duplicated with chemically produced oxides of nitrogen, used in the absence of the other gases usually found in normal atmosphere. This information prompts the thought: what is the cause of their presence in the atmosphere, can this cause be corrected and can they thus be eliminated?

It has been established as an indisputable fact that small quantities of oxides of nitrogen are formed when coal, oil and gas are heated to ignition or burned as we usually describe it. Goodall[1] states that the color changes have been produced in test apparatus by replacing the gas burner by an electric heating element. While that may be a theoretical possibility, it is doubtful that the use of portable electric heaters is the source or cause of the formation of oxides of nitrogen in quantity which will cause objectionable color change, even if the heater is used in a relatively small confined space.

An ordinary one-section electric hot plate with an open exposed coil was enclosed on four sides with metal sheeting to prevent escape of the heated air. At a distance approximately twelve inches above the coil, acetate satin, colored with two very sensitive dyes, was suspended for a period of two weeks continuously. During this period the temperature around these samples was reasonably constant and nearest the coil, between 180° and 185° F., and most distant from it, 10 degrees lower. During this time the coil remained between red and white heat and the position of the samples was changed twice daily.

Anticipating a possible influence by the high temperature, identical samples were exposed continuously in a steam heated oven-drier for four and six day periods at a temperature between 220° and 240° F. Very little color change was caused by the exposure over the coil and much less than by the heat in the drier.

The exhaust gases of automobile engines was next examined as a possible source of atmosphere contamination. The side of a muffler was cut open in a form that would cause the gases passing through it to escape in a known direction and within a known area. A 30 H.P., 6 cylinder auto engine, mounted on a block and running at a r.p.m. that would propel an auto at a speed of sixty miles per hour on level road, was connected. The fuel was a regular commercial grade of gasoline containing tetra ethyl lead. Two sets of acetate satin, colored with eight dyes of known variation in sensitivity, between very poor and non-sensitive, were suspended in a position where they would be in continuous contact with the escaping exhaust gases. The engine was run for 33 hours continuously when one set of samples was removed and the other exposed for another 33 hours continuously, a total of 66 hours. The temperature around these samples was between 190° and 200° F. They could not be exposed at a sufficient distance from the muffler to reduce the temperature without probable interference with continuous contact with the gases. Equipment can readily be arranged to correct that condition, however, it was not considered necessary at this time.

Anticipating possible influence of the high temperature, similar sets of samples were exposed in an enclosed coil electric heated oven for periods of 33 and 66 hours at a temperature between 195° and 200° F. thermostatically controlled. The samples colored with the known sensitive dyes exhibit readily observable color changes. However, the degree of color change is not more than approximately 1/10th of that which would occur in similar periods of time in a test apparatus with the burner consuming 2 cu. ft. gas per hour and with the temperature within the area of exposure between 100° and 110° F. Some of this color change may have resulted from the brushing and rinsing in water used to remove the film of lead salts remaining from the gasoline. It is possible, but yet to be definitely established that auto engine exhaust gases are responsible for more than a very small portion of the oxides of nitrogen found in the atmosphere. The samples treated in the oven exhibit no color change. An analysis of an average sample of auto engine exhaust gases discloses its composition as:

Carbon monoxide	7.00%
Carbon dioxide	8.96%
Hydrogen	3.00%
Methane	1.00%
Oxygen	1.46%
†Nitrogen	78.58%

*Presented at meeting, New York Section, May 24, 1940.

**E. I. duPont de Nemours & Co., Inc.

†The latter determined by difference and assumed to be in inert form. (See reference note [1].)

The atmosphere is known to contain appreciable quantities of sulfur dioxide and smaller quantities of the trioxide in areas where large quantities of coal is burned in manufacturing plants and homes. An acetate satin, colored with a very sensitive dye, was suspended for four days continuously in dry sulfur dioxide gas and another sample for seven days continuously in moist dioxide. During these periods the gas was changed daily.

In response to an inquiry whether refrigerant gases escaping from air conditioning and temperature conditioning units had color destroying influence, acetate satin, colored with a very sensitive dye, was exposed for six days continuously in a pure dry refrigerant gas at room temperature and then for five days longer continuously in the gas to which moisture had been added. During these periods the gas was changed daily. We were informed that the gas used is identical or similar in chemical constitution with that used in approximately 70 per cent of the refrigerators and air conditioning systems at present in use. Another sample of the colored acetate was exposed for several days continuously in a bottle containing a liberal quantity of 28 per cent liquid ammonia. This series of experiments illustrates that the color has remained unaffected by these treatments.

Information about the individual dyes applicable to the acetate fibers in relation to their resistance to atmospheric fading and in relation to their application properties is too well known to the technicians of their manufacturers and to the mill chemists and dyers to require comment by this author who doubts that he could provide additional useful information. Two members of this association, Dr. Meili and Mr. Cady[3], have prepared papers on the subject and their remarks are available in technical publications. In these reports are references to the reports of other investigators and where these may be located in technical literature.

Considered as classes or groups, it is well known that dyes which are diazotized and developed after application to the acetate fibers, exhibit the best resistance to atmospheric fading. Next best are the azo class of dyes, many water soluble and applicable in a one-bath operation. The most sensitive, in general, is the class usually described as the amino anthraquinones, which are applied in a dispersed form.

Being familiar with the chemical constitution of these classes of dyes in a general sense or way, technicians and chemists may believe that because a dye does not contain unsaturated or free amino groups that that alone is satisfactory evidence that it is not sensitive to atmospheric fading. This is definitely an incorrect assumption, because there are very few dyes which, after application to the acetate fiber, are definitely not affected by prolonged exposure in an atmosphere containing oxides of nitrogen. Some exhibit a definitely objectionable color change when subjected to test procedure under conditions that this author believes are well within the limits that a colored fabric should withstand without color change, if that fabric may be expected to exhibit satisfactory resistance during its useful life in general average use and/or storage. Exhibit D is test apparatus exposures for atmospheric fading of acetate satin colored with dyes that meet these conditions and includes dyes generally known as of the acid, basic and azo classes. Some exhibit color change, some loss in tinctorial strength and some both.

As of possible interest to those present that are not familiar with color problems, Exhibit E is acetate satin colored with two dyes of the developed class that produce blue shades, which satisfactorily withstand average atmospheric fading requirements, but which exhibit relatively poor, and probably unsatisfactory, light resistance. It must, however, not be assumed from this exhibit that every dye of the developed class exhibits poor or unsatisfactory light resistance. This exhibit is principally to illustrate the tremendous shade change that occurs as a result of development and how very difficult it is to use this class of dyes for accurate shade matching.

Being familiar with the cause of atmospheric fading, gave the chemist an indication of what could be done to counteract or avoid it. Many chemists of all the dyestuff manufacturers have for several years been endeavoring to produce new dyes that are not sensitive to atmospheric fading. This form of solution is not considered to have met all practical requirements satisfactorily at this time. There is available at least one blue dye that meets average atmospheric fading requirements and it is being used in considerable quantities, however, some dyers maintain that it cannot be used for all types of fabrics because of application problems which are responsible for increasing the cost for dyeing beyond what the selling price of the finished materials permit. Research chemists have also found substances which can be applied to the acetate fiber after coloring and which when properly applied impart a resistance to atmospheric fading which meets average requirements satisfactorily.

Exhibit F is two large pieces of a grey shade crepe dress material composed of acetate fiber and viscose process rayon. To the upper large piece is attached seven smaller pieces. The one to the extreme left of these was treated in practical dyehouse processing[4] and the remaining six were treated by the author, all with different substances at present available from three different sources of manufacture to provide protection against atmospheric fading. To the lower large piece is attached identical smaller pieces, but these have been subjected to test apparatus exposure for atmospheric fading. In this group, one extra piece has been added, attached to the extreme left. This is an untreated piece to serve as a control and to illustrate that the substances with which the remaining pieces have been treated have actually imparted a very valuable degree of protection. The untreated control has been changed very much redder, while those treated have been changed only very slightly greener and/or duller and these changes

would not be objectionable if they occurred uniformly distributed over the entire surface of a garment. It must also be remembered that by far the largest portion of this visible color change occurred when the substances were applied to the dyed fabric and not during the test exposure. It should also be remembered that the lowest row of small pieces has been exposed to a test procedure more color destructively severe than the average garment would meet during its practical useful life.

While these substances impart a degree of protection against atmospheric fading that will meet average requirements during the average useful life of a garment and under normal conditions of fabrication into garments they should possess that protection when they reach the consumer or customer, we must consider what may happen during their use by the average person. It will probably come into contact with perspiration, if it is a dress or lining cloth, and the owner may wish to have it dry cleaned or washed if it is a dress, and if it is a lining cloth, the gentleman may send it to the tailor for pressing and/or dry cleaning. To determine what, if any, influence these conditions would exert on the protection imparted against atmospheric fading, the author selected six different substances that impart satisfactory protection against atmospheric fading and applied them to acetate satin colored with a dye sensitive to atmospheric fading. Three of these substances are at present in use or available for use to the dyer and finisher for this purpose and the other three are not, to the knowledge of the author. This difference is not of particular importance for the present purpose because all are affected similarly by the treatments to which they were subjected, as are many other substances which have been evaluated experimentally by the author and found to impart a valuable degree of protection against atmospheric fading. After the application of the six atmospheric fading inhibitors, separate pieces of the material were subjected to:

A—3 treatments in carbon tetrachloride.
B—3 treatments in perchlorethylene.
C—3 treatments in trichlorethylene.
D—3 treatments in Stoddard solvent.
Each treatment for 15 minutes at room temperature and each treatment followed by drying in the air.
E—Saturated with the A.A.T.C.C. acid perspiration test liquor and dried in the air.
F—Saturated with the A.A.T.C.C. acid perspiration test liquor, dried in the air and then saturated with the A.A.T.C.C. alkaline perspiration test liquor and then dried in the air.
G—Saturated with the A.A.T.C.C. alkaline perspiration test liquor and dried in the air.
H—Saturated with the A.A.T.C.C. alkaline perspiration test liquor, dried in the air, then saturated with the A.A.T.C.C. acid perspiration test liquor and then dried in the air.
I—Treated for 20 minutes at 120° F. in an 0.5 per cent neutral soap solution, rinsed in warm water and then dried in the air.

Pieces of all were then subjected to test apparatus exposure for atmospheric fading and for three different periods of time.

Exhibit G is this series of experiments which illustrate that in every instance the treatment in the soap solution has removed the original protection against atmospheric fading that the inhibitors imparted. In every instance the perspiration liquors have also reduced the degree of protection originally imparted to acid fading. It is observed that there is an appreciable difference in the degree of the destructive influence exerted by the perspiration liquors on the materials treated with the different substances, indicating that some resist the destructive action of perspiration better than others. However, it must be remembered that this test procedure merely illustrates what may be anticipated as the effect of one contact with perspiration and not several which would be more nearly comparable with what the garment may be subjected or exposed to during its useful life[5].

This is a suitable point at which to direct attention to the fact that because these inhibitors are not foolproof, figuratively speaking, that they are not useful. They are definitely useful and unquestionably as their deficiencies become apparent and known, effort will be directed to correcting and eliminating them.

Acetate fibers are frequently used in combination with rayon for crepes. Obviously, if so combined the rayon would be subjected to inhibitor treatment if the acetate was. Colored rayons do not exhibit atmospheric fading tendencies. To determine if the inhibitors exerted influence on the principal fastness properties of dyes used for coloring rayon, an all viscose process rayon crepe was colored in relatively light or weak and also in medium depths of shades with 36 direct dyes which exhibit better than average light resistance when applied to that fiber and to cotton. Portions of these pieces were padded with 2 per cent solutions of five and of the six inhibitors used for Exhibit G allowing the cloth to retain its dry weight of the solutions. After drying at a relatively low temperature individual small pieces were:

A—Saturated with the A.A.T.C.C. acid perspiration test liquor and dried in the air.
B—Saturated with the A.A.T.C.C. acid perspiration test liquor, dried in the air and then saturated with the A.A.T.C.C. alkaline perspiration test liquor and then dried in the air.
C—Saturated with the A.A.T.C.C. alkaline perspiration test liquor and dried in the air.
D—Saturated with the A.A.T.C.C. alkaline perspiration test liquor, dried in the air, then saturated with the A.A.T.C.C. acid perspiration test liquor and then dried in the air.

These pieces, together with a control (not saturated with the liquors), were then pressed with an electric iron heated

to a temperature of 150° F. for a period of 20 seconds. This is Exhibit H which cannot be shown because when sewn to six inch width cotton cloth the length of the entire exhibit is in excess of 180 yards. Briefly described, the results illustrate that the different inhibitors impart some degree of sensitivity to both perspiration and to dry heat to some of the dyes. All of the dyes do not exhibit sensitivity when treated with all of the inhibitors, and each of the individual inhibitors sensitizes individual dyes. However, so few of the dyes are objectionably sensitized by any one inhibitor that no great difficulty should be experienced in selecting a range that would meet practical requirements for use with any of the inhibitors for this particular reason alone.

Some of the inhibitors objectionably affect the shades as well as reduce the light fastness of some or all of the dyes used for this group of experiments. This is illustrated by Exhibit I from which may be observed that three of the inhibitors reduce the light fastness of all or very nearly all of the dyes. Fortunately, two of these three inhibitors are not in use or offered for use, to the knowledge of the author. The remaining three are offered for use, however, the one of these that causes objectionable color change and reduces the light fastness objectionably for general purposes is offered with definite instructions that it is not recommended for application to materials that contain other than acetate rayon. One of the remaining two causes only very slight color change and only very slight light resistance reduction and should therefore, meet all reasonable requirements in these respects. The fifth and last for consideration causes objectionable shade change and light resistance reduction on some of the dyes, but not on all and it is, therefore, probable that a selected range of dyes is available with which this inhibitor can be used. The inhibitors used on the second and third from the top samples on the individual cards that compose this exhibit actually improve the light fastness of the acetate fiber dyes. These are the two inhibitors in practical use.

The author has been actively engaged in experiments with atmospheric fading test apparatus for at least seven years and because of his activity as a member of the A.A.T.C.C. Research Sub-Committee investigating test procedure and apparatus, he has received numerous inquiries regarding the subject. This has resulted in his having become familiar and acquainted with a number of those now engaged in testing. The courteous, kindly helpfulness of these gentlemen is hereby acknowledged with sincere thanks, as is their permission to use the information they have imparted in the following part of this address, the experiments for which were undertaken and are continuing by the author as his contribution to the A.A.T.C.C. Research Sub-Committee activities.

One of the first activities of this Committee was to prepare a number of samples of acetate satin colored with dyes of known varying sensitivity to atmospheric fading and submit them for test to eight persons who were known to be testing and using test apparatus designed by them for this purpose.

Briefly the results disclosed that while both the apparatus and test procedure differed in detail, all used illuminating gas as the means to provide the oxides of nitrogen. They differed principally in the time each required to secure a predetermined degree of color change or destruction. This indicated that all the apparatus could be used to provide the desired information if some means could be found to have all use an identical yardstick or system for measurement and description of sensitivity. It was then decided that a start toward that goal would be achieved if a set of standards exhibiting definite variations in sensitivity could be found and adopted. By that means if all used these standards, the results could be described in relation to them and thus tests made in any apparatus could bear a definite and fixed relation to tests made in any other apparatus. The ideal standards would be three or four in number, the second least resistant to show no color change when the lowest or most sensitive showed a definite one. The third to show no change while the second did and the fourth none while the third did.

We tried many dyes applied in different tinctorial strengths to achieve this object but were unsuccessful unless different colored standards were used.

This appeared inadvisable to the Committee until it was definitely determined that a range of blue colored standards made by combining a sensitive and non-sensitive blue dye in different proportions could not be made. This work is now under way. However, in connection with this work, we learned that there are very few dyes that do not undergo some change when exposed in test apparatus for periods of time and under conditions required to be comparable with what is believed a fabric should withstand to show satisfactory performance in actual use and/or storage. Some of these experiments are displayed for inspection as Exhibit J and they include material colored with acid dyes, basic dyes, water soluble dyes and dispersed types now in general use.

Test apparatus in use differ in some details to an extent that prompted the author to undertake some experiments to determine the influence of variations in temperature, gas consumed, air flow, humidity, time and other factors. These are offered for inspection as:

Exhibit K—variations in gas consumption of 2—4—6 cu. ft. per hour with temperature constant at 76°-80° F. and air flow at 3.5 cu. ft. per hour.

Time variations of 5—9 and 25 hours with gas consumption constant at 2 cu. ft. per hour, air flow at 3.5 cu. ft. per hour and temperature between 76°-80° F.

Variations in air flow of 3.5-7.0 and 14 cu. ft. per hour with other conditions constant and as described.

Dehydrated air containing the oxides of nitrogen and air with a relative humidity of 55-58 per cent at 76°-80° F. with all other conditions constant and as described.

These illustrate that variation in any of these factors

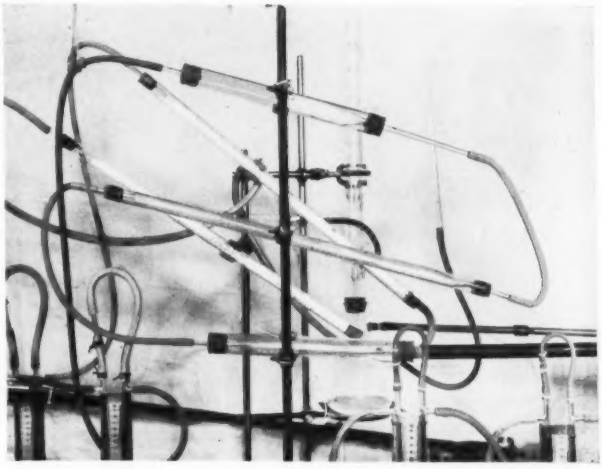
influences the degree of color change from slightly to very, very much.

Exhibit L—which consists of four cards, illustrates that a box with no mechanical means to aid air circulation and depending on the natural tendency of warmer to replace cooler air and thus cause air movement, can be satisfactorily used for testing if the position of the exposed samples is changed often enough, and that in such a box absolute destruction of a sensitive dye can be achieved with the consumption of only 2 cu. ft. gas per hour, if the burner is placed sufficiently near the means of entry into the box of the gas contaminated air, to raise the temperature within the box to 140° F. This exhibit also illustrates the tremendous increase in color destroying action that will result from having the burner heat to red heat a wire gauze through which the air must pass before it reaches the exposed samples[6].

The author has heard suspicion placed on paint fumes to cause color changes similar to those that are attributed to atmospheric fading. Exhibit M are acetate samples colored with a very sensitive dye which have been exposed in tightly closed glass tubes for 66 hours at room temperature while air at the rate of 3.5 cu. ft. per hour through a six inch diameter glass funnel inverted over the top of a quart can of a commercial grade of well known brand white linseed oil and turpentine paint was drawn through them. The other sample was exposed to the fumes from a Dulux hydrocarbon solvent type of lacquer under identical conditions. These treatments have not caused more than a trace of color change.

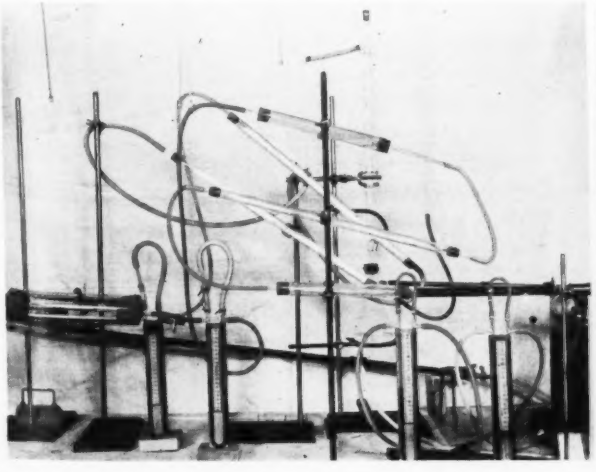
An experiment to determine the rate of color destruction caused by trapped air contaminated by oxides of nitrogen is under way, but shows no color change of any consequence after 72 hours, with change of the contaminated air at 12 hour periods during this interval.

The slides now shown as Exhibits K, L and M are for the sole purpose of illustrating the apparatus used for the experiments already discussed and displayed under identical designations. Slides K 1 and 2 illustrate duplicate apparatus arranged to determine the influence of moisture or humidity on the rate of color destruction. These experiments were not successful because no means was found to cool the contaminated air without causing condensation of the moisture in the tube in which the samples were exposed. It was obviously necessary to have the temperature in both comparable experiments uniform to secure the desired information. The condensed moisture is readily observable in the tubes through which the air was drawn in an effort to cool it. The third slide K3 illustrates in detail the glass tubes in which the samples were exposed. The fourth slide K4 illustrates the arrangement of the burners which provided the oxides of nitrogen contaminated air. The fifth and sixth slides L1 and 2 illustrate the box used for the experiments displayed as Exhibit L. Slides 1, 2 and 3 illustrate the apparatus constructed by the author and used by him for general comparative exposures. It has a capacity of 400, 1¼ by 2½ inch

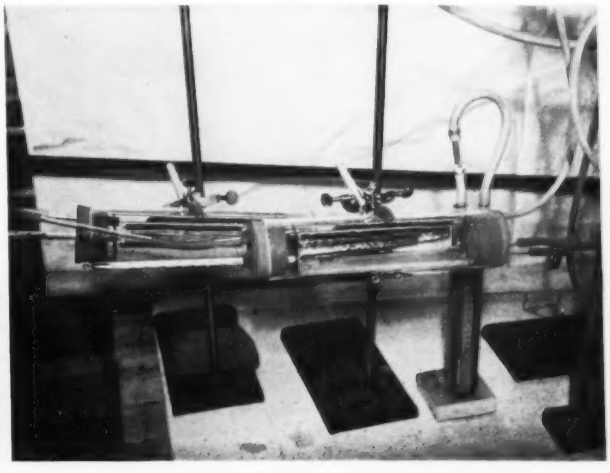


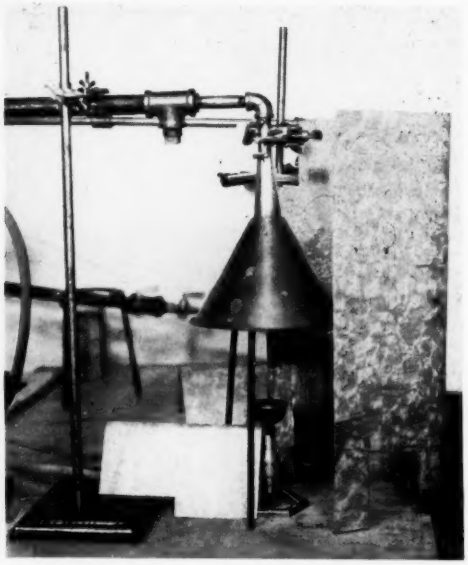
Slide K-2

Slide K-1

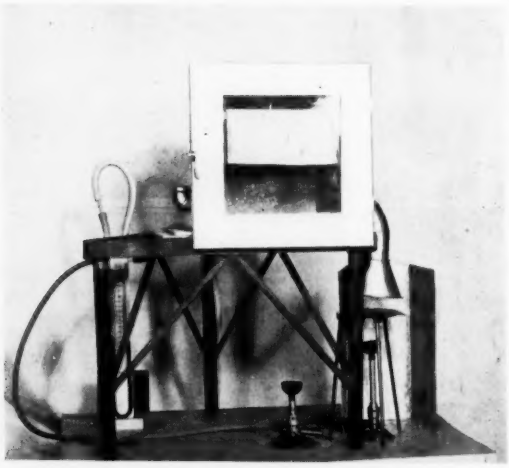


Slide K-3

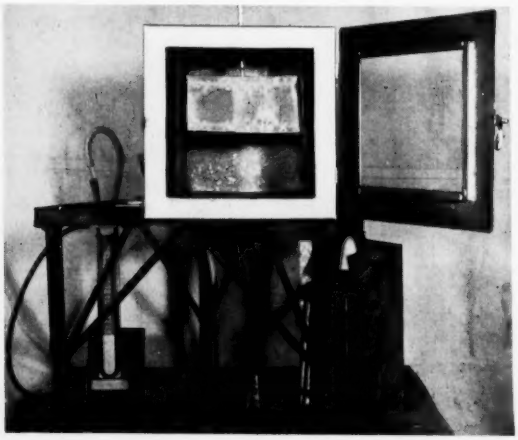


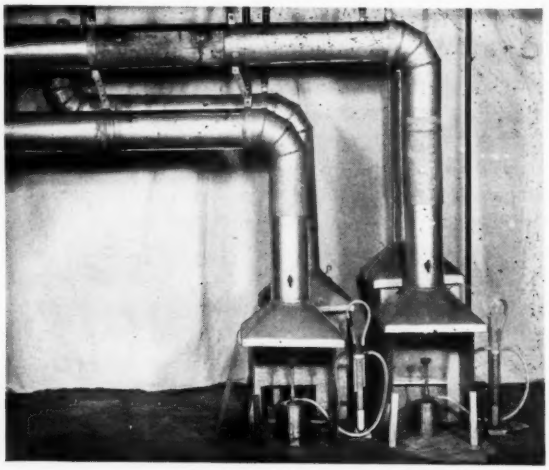


Slide K-4

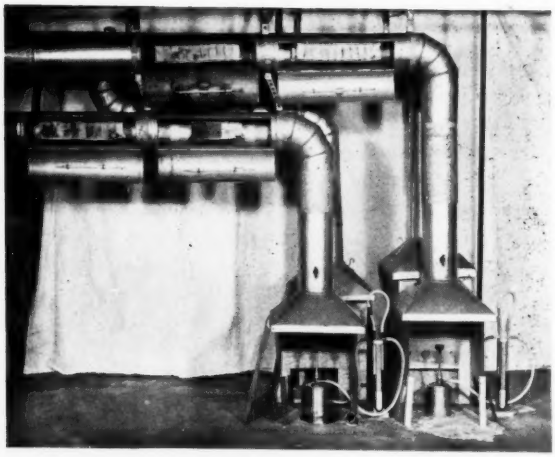


ABOVE—Slide L-1; BELOW—Slide L-2



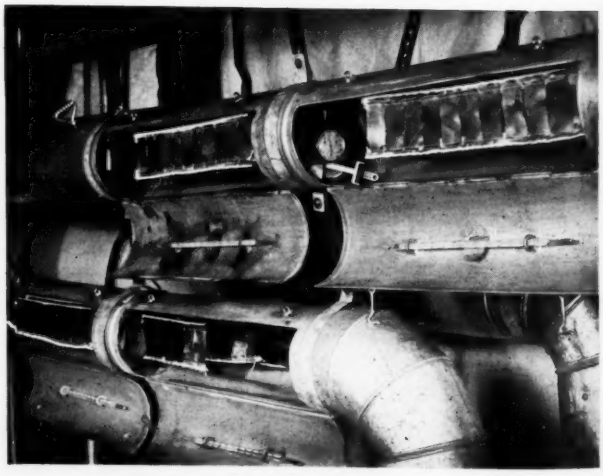


Slide 1



Slide 2

Slide 3



samples and this could be easily increased. These slides illustrate the use of manometers for gas and air flow control and of an anemometer to measure the air flow in the larger apparatus in which the flow can be varied between ten and several hundred cu. ft. per hour. Dampers are inserted to control the air flow which varies and must be controlled because the outlet is through a chimney, the outer opening of which is about 40 feet above the ground level.

Two of my friends who are engaged in testing, kindly provided drawings of the apparatus they have designed and are using. Photo A[7] illustrates an apparatus in which

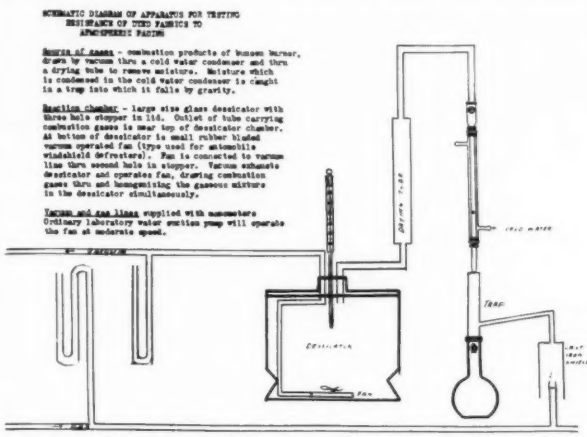


Photo A

the air is dehydrated before it reaches the samples being exposed. Photo B[8] illustrates an apparatus in which the

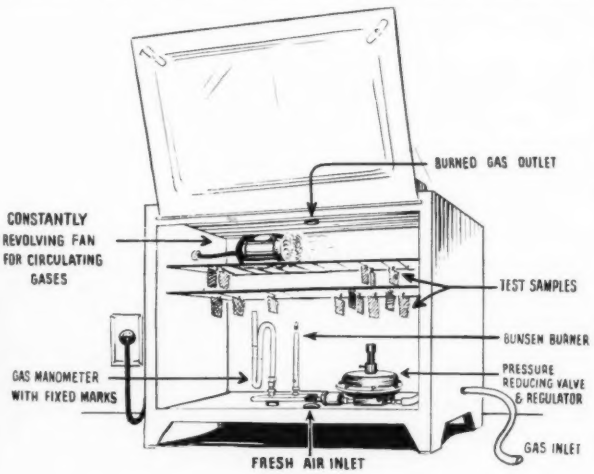


Photo B

dehydration system is not used and while the author prefers air containing natural humidity for testing purposes, he does not yet possess evidence that either system will not provide equally satisfactory results in relation to consistent reproducibility. However, those who have endeavored to check the consistent reproducibility of any apparatus designed to accommodate a large number of samples have learned that some means to cause uniform air movement and distribution within the apparatus is a definite necessity. Smaller apparatus can be used without this aid if the position of the samples is changed sufficiently often; at periods of no longer than 12 hour intervals, when the total test time is not for less than 48 hours and at 6 hour intervals when the total test time period is not less than 24 hours.

We know that wool and silk must be protected against the destructive action of moths and other vermin and that the substances at present used for this purpose do not continue to impart that protection for an indefinite time or under all conditions to which such fibers are exposed and subjected during their useful life and we, therefore, recognize the condition and utilize the valuable properties of these substances regardless of their limitations. Why not, therefore, use the inhibitors for atmospheric fading where they serve a useful purpose regardless of their limitations. Because the acetate fibers will not meet every condition under which we would like to use them or withstand every abuse to which we would like to subject them, does not appear to the author a valid reason why we cannot and should not utilize their very valuable useful properties and endeavor to exercise a reasonable care in their use so as to protect them from exposure to conditions which we know are harmful. If we utilize to the best advantage the dyes most resistant to atmospheric fading, use the inhibitors where they are valuable, dry clean and not wash acetate fiber fabrics, develop an inhibitor that can be applied by spraying or in gaseous form similar to present procedure for moth resistance and place some confidence in our ability to improve and correct present deficiencies, we will soon realize that the problems the acetate fibers present commercially are not insurmountable.

In conclusion, the author acknowledges with sincere thanks the valuable aid his assistant, Mr. Charles A. Sylvester, has contributed to the work that made this paper possible.

References and Notes

[1]Goodall—Journal Society of Dyers & Colourists, 1935, 51, 126.

[2]Since the preparation of this paper, the author's efforts to find an analytical procedure for the determination of the oxides of nitrogen present in the atmosphere and in test apparatus has resulted in his learning that there is some information available which indicates that the exhaust gases of auto engines may be responsible for the presence in the atmosphere of greater than suspected quantities of the oxides of nitrogen. The investigation to learn the facts is being continued and the information resulting will be included in a paper to be presented later.

[3]Cady—American Dyestuff Reporter, 1939, 28, 333.

[4]By the Hellwig Silk Dyeing Co. of Philadelphia, Pa., and made available to the author through the courtesy of Mr. Jacob Knup.

[5]During the display of this group of exhibits, the author included some comments not in the paper in order to direct attention to some particular point. After the conclusion of the address and discussion which followed, Dr. E. Freedman courteously directed the author's attention to the fact that the author had stated that perspiration was secreted alkaline and changed to acid as a result of decomposition, whereas the reverse is correct. The author gratefully acknowledges this correction with apologies to the audience.

[6]The author acknowledges the courteous helpful co-operation of Mr. David Seiferheld of the N. Erlanger Blumgart Co. for directing his attention to this fact.

[7]Apparatus constructed by Herbert Schenker of the Consumers' Testing Laboratories of Philadelphia, Pa.

[8]Apparatus constructed by Charles L. Simon and his colleagues of the Industrial By-Products & Research Corp. Gimbel Brothers of Philadelphia Bureau of Standards.

DISCUSSION

Mr. Freedman: Mr. Chairman, I would like to start off by saying that Mr. Seibert's presentation of his paper this evening discloses the fact that he has conducted an investigation that is bound to have a very salutary effect on not only the dyeing industry, but also probably on the dyestuff industry, the finishers, the textile converters, garment manufacturers, retailers and consumers.

I would like to discuss, if I may, a number of points that Mr. Seibert brought out this evening and probably during my discussion point to a rather different angle as an approach to this subject.

First of all, I would like to say that, from the point of view of the consumer—up to the present time—inhibitors are not satisfactory, because of the effect on them of perspiration which, when freshly exuded, is acid. Incidentally perspiration in the process of decomposition turns alkaline but meanwhile the acid perspiration has brought about a neutralization of some of the inhibitors used on dyed fabrics, after which the fabrics discolor, the blue dye reddening.

First of all, garments must be washed or dry cleaned. Many of them have to be washed, because certain soils or soiling agents contacting the garments are not removable by dry cleaning fluids. Also, dry cleaning is not at all times truly a dry cleaning process. It also includes a wet wash.

A consumer buying a garment or a fabric that proves satisfactory until it is cleaned will not remain satisfied if there is a change in color after the cleaning process. Up to the present time, many consumers have been led to believe that the change of color has been due to abnormal perspiration or due to the use of deodorants or antiperspirants.

While that has been true in many cases, we have run across instances where the opposite has been true, where the type of reddening found in the garments returned by consumers, found in draperies, was reproducible by a laboratory gas fading test. When I say reproducible, I mean that both the intensity and type of discoloration, appear to the eye to be the same.

So I don't think that inhibitors are quite the thing. As a matter of fact, inhibitors have been furnishing an alibi for many people, because fabrics have been coming upon the market, have been stored, made up into garments, sold, while only after the garment has been subjected to perspiration or has come in contact with water or has been washed, or at times dry cleaned, has a fabric faded.

The day of judgment has been passed along to a point where the placing of responsibility has been rather difficult, and considerable controversy has arisen. If inhibitors have to be used and if there is only one good blue dyestuff available, then it appears to me that the merchandise so dyed need not necessarily be marked, but it can be marked or labeled so as to disclose that it will resist atmospheric fading.

However, it also seems no less proper that merchandise that is not capable of resisting atmospheric fading should be marked at the source of manufacture by the dyer to disclose the fact that that merchandise will fade so that the purchaser, be he either the converter, the garment manufacturer, the retailer or the consumer, shall be informed of what to expect.

We have been using in our laboratory a number of methods in an attempt to solve this problem of atmospheric fading, and until recently we were getting all sorts of results. But not so long ago, through the liberal contribution of the Better Fabrics Testing Bureau, we received a gas chamber similar to the one displayed. I believe it was Exhibit L. We used a burner similar to the one displayed in that exhibit, a Tirrell burner I believe it was, with a wire net placed over it.

We placed a cylinder or shield around the upper part of the burner so as to protect the flame against draft. We used a colorless or a blue flame. We used sufficient heat or sufficient gas to bring about and maintain a temperature of 170 degrees Fahrenheit in the oven.

The top, the rear and the far side of the oven were insulated with quarter-inch transite so as to help maintain the temperature of the oven at about 170 degrees, plus or minus three degrees, and also to insure that the far side of the oven would not cool off or that the temperature there would not be much less than the temperature at the near side. The oven was equipped with a baffle plate.

We were able to take garments returned by customers for atmospheric fading and reproduce the type of fading in many cases by an eight-hour fading test and in other cases by a sixteen-hour fading test.

It seems to me that at 170 degrees Fahrenheit and running the test for sixteen hours, we have at hand for the present a test method that permits us to determine whether or not our merchandise is going to prove satisfactory in resistance to the NO_2 fumes in the air.

I agree that circulation of air is very important, and I believe that further investigation will bring about refinements of the test methods.

I cannot tell you how serious this problem is in the retail field. I can say that the Retail Dry Goods Association of New York has held a meeting with the manufacturers of the acetate rayon and they are going to hold more meetings. I think the time will come very shortly when we will ask those in the dyeing and finishing industry to tell us, through their converters, whether or not the materials that have been treated with inhibitors are going to fade, so that we can buy the merchandise with our eyes open and pass that information on to the

ultimate consumer in order that she in turn can buy that merchandise with her eyes open.

I have one more remark and then my discussion is over, and that is this: I would like to sound a word of caution insofar as the use of inhibitors is concerned.

The skin is normally acid. The use of inhibitors of an alkaline nature may affect the skin and increase the likelihood of bacterial invasion, which may bring about either dermatitis or infection.

Chairman Meili: Well, I don't think that Mr. Freedman's talk needs any answer. It is enlightening in itself.

Are there any other questions to be asked?

Mr. Kapelsohn: Mr. Seibert, in connection with the conduct of the test using the gas flame and especially when a wire gauze is used over the gas flame, it seems that there are a number of factors leading to possible error that have not yet been investigated.

For one thing, we have no control over the composition of that gas, and I believe that it does vary from time to time, depending upon both the locality and the conditions in any one locality.

Secondly in using a wire gauze over the flame you find a greatly accelerated effect. But how does that vary as the gauze becomes older? Is that a catalytic effect of the wire gauze increasing the production of oxides of nitrogen or is it merely the effect of increasing the heat because the wire is there, and how do those two factors affect the accuracy of the test? Can anything be done along those lines?

Mr. Seibert: Some yes and some no. I would like to answer the last part of the question first by saying that in these experiments the temperature was definitely controlled; in other words, it was definitely 120 plus or minus one.

What influence continuous use of the wire gauze has, I do not know.

We made an intensive effort to develop a method of analysis whereby we could determine definitely the quantity of oxides of nitrogen present in the test apparatus. Those efforts are still continuing. I have some figures; however, I purposely did not include them this evening, because we have not developed the method, sufficiently controllable in reproducibility, to feel that it has practical value yet.

Obviously, if we could perfect a simple method for determining the quantity of oxides of nitrogen in the air, it would be the best control for testing that I can think of, and I believe in the near future something of that sort will be available. It isn't at the moment.

In relation to the difference in the composition of gases furnished for illuminating purposes in different localities, it is doubtful that they vary sufficiently to have an influence on the results that will cause those results to be unuseful. I believe that there is very little difference between them for test purposes, because in the original experiments that were made by the Atmospheric Fading Tests Committee, two were made in Rhode Island, two in Pennsylvania, two in New York, and two in New Jersey.

That is relatively Eastern states only, but it may be a fair cross-section of the gas that is furnished throughout the country.

May I add that one of those gases was a natural gas, Pyrofax, and in no instance did we find a difference in those tests that was significant. True, they did vary in the degree of the destructive action in a specified length of time, but at some point every apparatus would show the same result.

Let us take as an illustration the most destructive tests which were the Erlanger, Blumgart and Industrial By-Products Research Corp.; Erlanger-Blumgart, fifty hours; Industrial twenty-four hours. They destroyed everything—well, not quite destroyed, but they reduced the non-sensitive dye about 30 or 40 per cent in tinctorial strength, while none of the other apparatus tests did that, or in fact caused any effect on the non-sensitive dye.

However, I feel confident that if we could in either one of these two apparatus make a test for a shorter length of time than was used for the tests made originally, we would get a result that was almost identical with any of the other apparatus that did not in their test procedure show as much destruction as these most destructive ones.

For test purposes variation in gas may cause minor differences, but it is extremely doubtful that the results they produce cannot be used as a reliable indication of what may be expected of any fabric during its average use in storage or under other conditions.

That is perhaps not answering your question, but it is describing to you our experience.

Mr. Kapelsohn: There is just one more point, which was brought up, that I would like to ask about. What attempts have been made to utilize the oxides of nitrogen either alone or in combination with sulfur dioxide as the source of the gas fading instead of going about it indirectly by using the gas flame?

Mr. Seibert: Do I understand your question correctly? Would the production of the oxides of nitrogen by chemical means serve the purpose?

Mr. Kapelsohn: Yes, control their production by chemical means?

Mr. Seibert: Yes. That was the first means used, taking a definite quantity of sodium nitrite, chemically pure, a definite quantity of hydrochloric or sulfuric acid. We knew that these definite quantities would produce a certain quantity of gas.

The nitrite and acid was placed into a No. 2 museum jar, followed by the test samples suspended above and the jars sealed. We found then that we did not have reproducibility. We very quickly found that the intensity of the light was a very vital influencing factor; in other words, the destructive action was directly proportionate to the intensity of the light, that is in relation to the time required to cause color change.

If I placed such a sample in diffused light, another in direct sunlight and another in the Fade-o-Meter, the Fade-o-Meter produced a reaction or any degree of destructive action very nearly the same as the others, however in a time a little faster than direct summer sunlight, and both very much faster than in the diffused light.

If you let them run long enough with the same definite quantity of oxides of nitrogen in the jar, chemically calculated, you got the same result, but it required different lengths of time. You could get the same increased activity of the Fade-o-Meter, by placing the museum jar in hot water.

This is an interesting and a most peculiar fact. We observed that if you placed the jars in the Fade-o-Meter where you could watch them very, very carefully, they would go on for a given length of time, probably 70 per cent or more of the time required to cause complete destruction before any change was observable, and then frequently in thirty seconds the color on the sample would be destroyed.

However, even in the Fadeometer it was very difficult to control the intensity of the light, because from a chemical standpoint, or as a chemist likes to control a reaction even the Fade-o-Meter is not a suitable apparatus, because it varies too much in its light intensity, as those who work with them will tell you, for a delicate test of this sort.

That method of procedure, therefore, was discarded as not being sufficiently accurate in reproducibility. By the way, that method is as old as I am, because I can remember, when I first went to work and learned the first thing about synthetic organic dyes, that method was used for the testing of the lining cloths then dyed with benzopurpurin, and dyes of that kind, which were sensitive to acid, and changed color during use and storage and the change was attributed to the causes at present under discussion.

They used that method merely as an indication in advance, when they could have gotten the same result by a controlled pH test with any indicator.

Have I answered that question?

Mr. Kapelsohn: I was thinking more of control of circulation of the gases.

Mr. Seibert: No. As I pointed out earlier this evening, I had a thought identical with yours; that is, I would like to know what influence, if any, the presence of sulfur dioxide and trioxide in the atmosphere exerts to activate the oxides of nitrogen, in color destructive action, but so far I haven't found a satisfactory controllable means. There is no question but that it can be done and I propose to do it.

Mr. Freedman: Mr. Seibert, I ran some tests along that line and I can confirm what you say about it.

Mr. Seibert: It is a difficult thing to control. It is not an easy matter to put them together in a simple controllable way, although it is not an impossibility.

Dr. Smith: I would like to ask if any tests have been run on the influence of finishing agents which might be commonly used with dyed acetate fabrics as accelerators of this fading.

It strikes me that many of the examples we see have faded so rapidly in comparison with the performance of the better dyes that there is a question whether some of the agents that are used as finishes might not be accelerating the trouble. I wonder whether any work has been done along that line.

Mr. Seibert: Dr. Smith, there hasn't been any by me, although I will promise you that there will be some done, because another gentleman this evening asked the same question. We will do anything that anyone can suggest within reason that promises to provide useful information.

I think you will agree with us, from what you know, that inhibitors are not foolproof, and you are not offering them as being foolproof. There may be other factors involved which we have not as yet discovered that are active influencing destructive agents and that isn't all we don't know.

I question whether we are justified in discouraging use of these fabrics at present; not at least, until we actually know what happens to them under controlled conditions in use and storage. We should know more than we know today.

Garments come back to Mr. Freedman. He is told certain facts about them, but he isn't always certain that those are the only facts that have contributed to the destructive color action. We don't have enough information yet to know just exactly how long it takes a sensitive dye under normal conditions in use and storage to be destroyed or show a color change.

Obviously, we anticipate that in a city like Pittsburgh it will occur much more quickly than it will occur in open country like that in South Jersey in which I live. We no longer make important sunlight and daylight tests in the plant, because we know the atmosphere contains abnormal quantities of gases and acids of all sorts, and they definitely do have a sufficient influence on the exposures to frequently make them misleading.

Chairman Meili: Dr. Smith, I can answer that question for you, that the finishes actually have an influence.

If you take the same dyed fabric finished with an acid finish, if it is a finish that is on the acid side, it will fade much quicker than one that is finished neutral or on the alkaline side. That probably answers your question.

Mr. Seibert: That is only natural, Dr. Smith. We anticipate it.

Mr. Freedman: Dr. Meili, I would like to say that we have the same experience. Here is a specimen of material, an original specimen. It is very small. This is the color change after eight hours in the oven at 170 degrees.

Here is the same specimen washed by the A.A.T.C.C. method wash test. The color change here and here is the effect after a standard dry cleaning test. There is less

color change after the A.A.T.C.C. method of washing and considerably less after the dry cleaning test.

Here is another case where the original specimen discolored considerably after being placed in the oven for a period of eight hours at 170 degrees, but there is practically no color change. There is just slight color change after the wash test and no apparent color change after the dry cleaning test, showing that the finishing agents or sizing used on materials does effect the color change of the dye.

Mr. Muller: I would like to bring out one point. It seems to me that, of all the dress troubles that the dyers here have, most of the dresses that come back to us dyers are really faded in the dress houses or in the stores.

If a dyer should use an inhibitor that would protect the goods as long as they are not washed or dry cleaned, to what extent would you think the dyer would protect himself? For instance, what percentage of dress claims come from the consumer and what percentage happens right in the dress houses in New York?

Mr. Freedman: I cannot give you any figures to disclose the percentages, but I would say this: That there are numerous garments being returned to retailers and that in some cases the garment manufacturers refuse acceptance of those dresses for return, but that really isn't the question at the present, as I see it.

As I see it, it is this: That the use of inhibitors merely delays the day of reckoning, because eventually that dress is going to fail. It may fail because of the action of the acid perspiration neutralizing an alkaline inhibitor put on the material.

It may fail as a result of dry cleaning or as a result of washing. If that takes place, then there is, to my way of thinking, a proper reason for complaint. So if inhibitors are going to be used, then I think the smart thing to do would be to label the merchandise with a blunt statement that this material has been treated to retard fading as a result of atmospheric conditions.

Removal of the finish by perspiration, washing or dry cleaning will accelerate the discoloration or fading of the material. If a finish is not used, then just make a statement, as we are trying to do now. We are telling our customers that the art of dyestuff manufacture and of dyeing has not yet progressed to a degree sufficient to give us blue colored acetate rayons that will resist atmospheric fading.

I think it is just a matter of days or maybe a week or so before a tag of that sort may accompany acetate rayon garments and other materials containing blue dyestuff that fade when subjected to test.

Mr. Chandross: I would like to add a word to what Mr. Freedman has been saying; that is, the day of reckoning is not very distant, and that the day of reckoning may be the first day that the customer wears that garment.

It must be remembered that perspiration is not confined to the armpits and that no garment that we in the department store laboratories have ever seen come back from the customer has failed to discolor at areas which directly contact the body at the shoulders, neckline, and other sections, which are not protectible by shields and other commonly used devices.

So that the day of reckoning consists, I believe, of merely passing the buck to many of the retailers who will have to make good to the customers, since we all have the garments fading in stock. It has been only after the first or second wearing, I believe, that there comes the first major objection to the use of inhibitors. It is granted that postponement of the day of reckoning will help many of us.

While I am on my feet I would like to ask a question of Dr. Seibert and that is, has the effect of moth gases or moth repellents, such as paradichlorobenzene or naphthalene, been studied as to their effect on acetate dyestuffs?

Mr. Seibert: Not to my knowledge. That gives us a thought for something else we can investigate.

Dr. Smith: I can say we bought a number of different moth repellents in New York about two months ago and put each one in a closed beaker with samples of acetate fabrics dyed with sensitive dyes and they haven't faded yet.

Mr. Blank: Mr. Chairman, while the retailers have the floor, I would like to add just a word about the extent of these complaints. We have member stores all over the United States. Practically all the department stores and chains are members, and we are getting complaints from every section of the country, and to such an extent that I believe there is a real danger that customers will begin to refuse to buy acetate rayons unless this thing is corrected.

We are very much concerned and interested in this subject that you are taking up tonight as to what we can tell our retailers all over the United States about this gas fading proposition.

Chairman Meili: Well, I still believe that if inhibitors are used, your complaints will be greatly diminished. They may not be entirely eliminated.

The garments probably will not fade before they are washed or dry cleaned. Even perspiration will not cause an immediate fading, it only makes the dyestuff more sensitive to fading, as Mr. Seibert has very well explained.

Mr. Blank: Are there any special inhibitors that can be recommended for that purpose?

Chairman Meili: There are several inhibitors on the market that are real value; they are very similar in their effectiveness.

Dr. Smith: Dr. Meili, I think there is just one interesting point that we ought to consider, too: acetate garments have been on the market for at least eight or ten years or more and, while the amount is increasing and while it is true that gas fading as such has only been recognized perhaps commonly for two or three years it would

appear that the retailers are getting many more complaints of the type of fading which is now called gas, and which may have been called perspiration before, than they did prior to a year or two ago.

It would seem to point to some condition that is different now than was existent previous to a year or so ago, and that is what makes me mention again the question of whether there are some finishing agents that have appeared or some step in the process of finishing that has appeared inadvertently which is influencing the rate of fading of these dyes, because the dyes themselves have certainly not gotten worse on the whole.

The assumption would be that our dyes are at least as good as they were. The atmosphere hasn't changed a great deal. All that has changed is the frequency of these complaints and the fact that complaints which are now known to be gas fading may have formerly been classified as perspiration or something else. But it seems to me that the stores would have been having all this trouble as other types of fading if the total number of complaints had been the same.

Therefore, there must be some factor which we haven't gotten at yet, which is increasing the frequency and the intensity of this thing. And I think for that reason we ought to go after it to study the different types of finishes or materials that are used, as we can see from this exhibit of Mr. Freedman's that in some cases the resistance to gas fading has been markedly improved by removing the finish or other material which was on the goods.

Chairman Meili: The dyestuffs used today are practically the same as were used eight years ago. I came in contact with gas fading about eight years ago and have heard complaints ever since.

The finishes, too, are practically the same today as they were at that time. Finishes are being used once in a while that should not be used. However, most dyers are fully aware that they should not use any acid finishes and they are very careful, though it may be that one dyer will make a mistake, but that will be an exception.

We have more complaints today because there has been more advertising of the problem. There is actually quite some hysteria about it.

Acetate fabrics treated with an anti-fume finish would surely pass the storerooms and the cutting up rooms without any difficulty. In fact, it is not only the blue dyestuffs that fade when exposed to gas fumes. Most of the other dyestuffs fade also. And you very rarely have any complaints about the yellows or reds. You only hear about the blues and sometimes about the violets.

If the blue and violet fabrics are treated with anti-fume finish they are about as fast as the other colors. So they should be sufficiently fast for normal requirements.

George Baxter: There is one radical change that takes place and that is the continual change towards lower prices. (Laughter). This may be a factor whether you are inclined to laugh at it or not. With the opening of dye houses in houses big enough to install two bathtubs, it is a natural thing for the use of the cheapest type of dyestuff in order to meet present-day prices.

Perhaps Dr. Smith or Mr. Freedman could give a better idea than I can of what percentage of fabrics, such as the acetate satin shown here tonight or acetate fibers, are treated with inhibitors, but it would be my guess that it would be less than 1 per cent of the fabric sold on the market.

Mr. Freedman: In answer to Dr. Meili's remark about color changes in fabrics dyed other than blue, we have run quite a number of tests and we are continuing to do so on all of the acetate rayons and mixtures containing acetate rayons coming up to our Bureau. We are finding very few specimens, other than blue, that are affected by our oven test method. We are, however, finding many of the blues that are incapable of withstanding the test without fading.

I have quite a number of cards here. I shall be glad to show them to you later if any of you care to see them.

ADDENDA

July 10, 1940

Since Mr. Seibert's presentation of his paper, Macy's Bureau of Standards has been conducting a series of tests in co-operation with Mr. Seibert. Along with the conduct of these tests certain refinements have been added to the gas-oven. As a result of these added refinements, Mr. Freedman finds that he can modify his previous opinion as to temperature. Previously, Mr. Freedman held that the temperature of the oven should be maintained at 170° F. because he found that the degree of fading at lower temperatures was (due to poor circulation and insufficient production of oxides of nitrogen fumes) not always easily discernible. With the refinements which are tabulated below, it has been found that a temperature of 140° F., a time factor of eight hours and a gas consumption of 2.2 cu. ft. per hour are capable of producing a readily discernible degree of fading.

Refinements to Gas-Fading Oven as Developed by Macy's Bureau of Standards

The Grid

Composition: Iron wire
Characteristics: 0.42 mm. wire diameter, 16 openings per inch
Size: 4 cm. square
Location: 3 cm. above burner top
Temperature: 730° C.—as measured by thermocouple

The Chimney

Diameter: 9 cm.
Height: 12.5 cm.
Distance of top from oven intake: 1 cm.
Bottom: 5 7 mm. diameter holes at base for air
(NOTE: A one pound sodium carbonate can proved satisfactory)

The Fan

Composition: Brass, painted aluminum finish
Size: 18.4 cm. length, 2.5 cm. width
Location: at center of bottom of chamber with motor underneath oven
R.P.M.: 200

The Burner

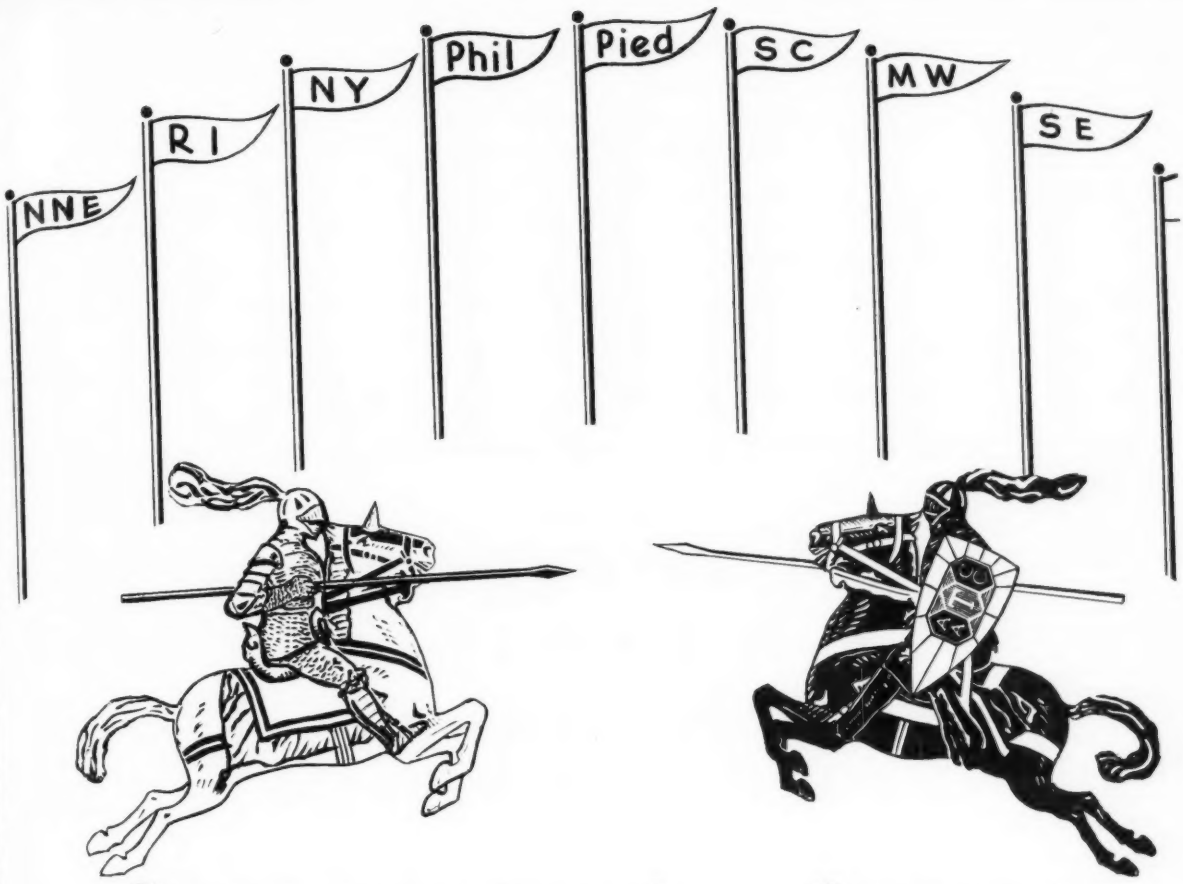
A No. 3 size Meker type burner, with Monel metal screen heat intensifier attachment, Eimer & Amend Cat. No. (1936)-19317

Temperature

140° ± 2° F.

Gas Consumption

2.2 cu. ft. gas per hour—average



Intersectional — Contest

FEATURING the technical program at the twentieth annual meeting and convention will be a contest in which each section will present a paper in competition with the other sections. These papers will be based on original, scientific or technical investigations of interest to textile chemists and colorists, either directly or indirectly. The contest is scheduled to be held on Friday afternoon, October 18th, and the names of the judges will be announced later.

Many of the sections are already at work preparing their papers, the titles of which appear below.

An award for the best paper presented will be made at a luncheon to be held at the New York World's Fair on October 20th, which has been set aside as a "Special Day" for this association.

Following is a list of titles submitted to date by the various sections:

Northern New England—"What is New in New England."

Rhode Island—"The Effect of Modern Finishing Agents on the Light Fastness of Cotton Colors."

Philadelphia — "Variables Encountered in Fadeometer Testing."

Piedmont—"Screen Printing in the South."

Southeastern — "Some Observations on the Tendering of Vat and Naphthol Dyed Army Duck on Total Exposure to Weathering."

Annual Meeting and Convention
October 18th, 19th and 20th, 1940

Unemployment Register

Members of the association who are without employment may obtain from the Secretary, or the American Dyestuff Reporter, application blanks for the Unemployment Register. Personal histories and employment records of applicants may be examined by prospective employers.

Twentieth

ANNUAL MEETING AND CONVENTION

OCTOBER 18TH AND 19TH, 1940

HOTEL COMMODORE

NEW YORK CITY

OCTOBER 20TH, AT N.Y. WORLD'S FAIR

General Chairman

PATRICK J. KENNEDY

Auspices of

NEW YORK SECTION

Make Your Plans Now to Attend!

TRADE NOTES • NEW PRODUCTS

• FIRE RETARDANTS

Loss of life and property damage from fire in suburban and rural communities could be substantially reduced by chemical fire retardants, Dr. W. E. Gordon of E. I. du Pont de Nemours & Company, said in an address recently before the Hudson Valley Volunteer Firemen's Association Convention at Haverstraw, N. Y.

In the last decade millions of Americans have moved away from cities to suburban communities where they depend for fire protection on volunteer fire departments. Suburban houses, miles away from fire hydrants, are frequently lost because fire consumes them before firemen can be summoned, the speaker said.

"Widespread use of fire retardants on draperies, clothing and insulating materials would greatly reduce these fire losses by preventing the rapid spread of the fire until the arrival of suburban fire departments.

"It is particularly important that inflammable insulating material be treated or 'flame-proofed' to prevent the spread of fire in frame dwellings. In some cities the building codes now prohibit the use of inflammable insulating material.

"The United States Government, which is sponsoring the use of cotton linters and waste cotton products for insulating batts or blocks to be placed between the upright studs in residential construction, has specified that such insulating material must be flame-proofed.

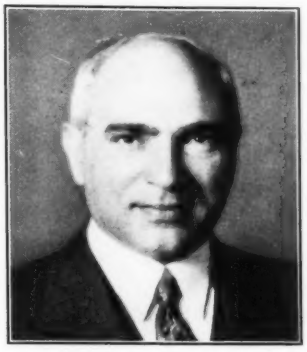
"It is now possible to flame-proof women's negligees, smoking jackets, sleeping garments, dresses, draperies and other interior decorations.

"Treatment of pile fabrics is particularly important since these fabrics when ignited will 'flash,' that is, the flame will sweep across the nap in an instant.

"Chemical research conducted in Du Pont laboratories has developed ammonium sulfamate, a chemical fire retardant which does not mar the original appearance of the fabric. This chemical will remain in the fabric indefinitely and will withstand dry cleaning. While laundering will remove the chemical, the dress or drapery can be retreated at a small cost."

• JOINS FOOTE

William A. Maxwell, who has just received his degree in Chemical Engineering, at the South Dakota School of Mines, has joined the research staff of the Foote Mineral Company, of Philadelphia. Mr. Maxwell, who has specialized in the chemistry of the less familiar group of elements as well as minerology, will devote most of his time to process development.



Victor H. Berman

• REAPPOINTED COMMISSIONER

Victor H. Berman, president of the Onyx Oil & Chemical Co., Jersey City, N. J., has just been reappointed for another term of five years as Commissioner of the Interstate Park Commission by Governor A. Harry Moore of New Jersey and Governor Herbert Lehman of New York. Mr. Berman was also re-elected Treasurer of the Commission for a five year term.

• A.S.T.M. TEXTILE REPORT

An extensive report was presented by Committee D-13 on Textile Materials at the Nineteenth Session of the Forty-third Annual Meeting of the American Society for Testing Materials held in Atlantic City, June 24 to 28. Included in the large number of recommendations were nine new tentative standards, some of outstanding importance, including Specifications for All Wool, All Cotton, and Wool and Cotton Blanketing (Household). This classifies blanketing into eight types and prescribes various physical requirements—yarn count, minimum weight, thickness, breaking strength and air permeability. Four new test methods and tolerances were given covering: glass yarn, woven glass tapes, woven glass fabrics and woven glass tubular sleeving and braids. The new standards give definitions and tolerances and cover such matters as breaking strength, weight, glass content and other properties.

There was considerable interest in two other new test methods—one involving the resistance of fabrics to water and the other resistance of fabric and yarns to moths. The scope of the first-named method indicates that they cover the following three procedures: hydrostatic pressure method, water penetration (drip) method, and water absorption (spray) method. The other method covers the procedures for determining resistance to moths of treated fabrics or yarns which contain wool or other hair fibers. The term "moth" shall be construed to include carpet beetles as well as clothes moths. Among other items specified are the type of larvae, average weight and the number to be used.

An important accomplishment in connection with numerous activities of the committee in the field of wool and its products was a new tentative test for shrinkage of grease wool which is intended to determine the shrinkage and wool content of samples of grease wool or individual fleeces on a laboratory scale. The method is accurate for tests on samples of the materials but does not necessarily mean that the results obtained on the sample are representative of a large lot.

The Society also approved on the recommendation of Committee D-13 revisions in quite a number of tentative standards and formal standards, some of those in the standards being adopted immediately. The requirements for Bleached Wide Cotton Sheeting (D 503) were modified by change in classification and the specifications and test methods for fineness of wool tops were also changed. Five existing tentative specifications, tests, and definitions were approved for reference to letter ballot for adoption as standard covering the following: Terry (Turkish) Toweling (D 505), Testing and Tolerances for Continuous Filament Rayon Yarns (D 258), Fiber Length of Wool (D 519), Wool Felt (D 461) and Definitions of Terms Relating to Textile Materials (D 123).

The report summarized some of the current activities under way in the committee in both the fields of standardization and research. All of the groups working on cotton, wool, asbestos, household and garment fabrics, glass fiber and its products, bleaching, dyeing and finishing have important projects under way.

• "PIPE BODY" CONSTRUCTION

The Rodney Hunt Machine Company of Orange, Massachusetts, announces a new, patented "pipe body" construction on its line of stainless steel reels for textile machines. The new feature is said to provide a greatly increased strength where it is most needed. Such reels are fortified against bending under heavy load pressure from the materials in the machine and also against twisting as the machines are stopped and started.

This new patented feature is claimed to have made it possible to build and equip larger dyeing machines than has heretofore been possible without the necessity of middle bearings on the reel. An example is a recent installation for the Botany Worsted in which a 20-foot stainless steel reel was built by Rodney Hunt without a middle bearing. They state that this is the largest

Trophy won by Mr. Smith. It reads "Atlas Ometer Trophy, Annual Golf Tournament, Mid-west Section, A.A.T.C.C."

Elmer F. Smith, (left) manager, American Aniline Products, Inc., Chicago, being presented with the Atlas Ometer Golf Trophy by V. S. Weiler, Sales Manager of the Atlas Electric Devices Company, Chicago, at the dinner following the tournament.

stainless steel reel ever built. Two 15-foot reels have also been built recently for the Hind and Harrison Plush Company.

It is said that this patented improvement in stainless steel construction of reels is of considerable importance to the trade as it enables building machines with much greater production capacity than heretofore. Many old style wooden dyeing machines are being equipped with stainless steel reels to save time in cleaning when changing colors.

• GENERAL RELEASE

General Dyestuff Corp., 435 Hudson Street, New York City, announces release of a circular describing:

Cellitazol BNF Extra Soluble; Cellitazol 3GNF Extra Soluble—two improved acetate soluble diazo blacks. It is stated that they can both be easily dissolved in hot water requiring no dispersion and which, for this reason, are said to give best penetration on piece-goods, yarns and tops. These powder brands will not dust nor tar upon boiling in concentration; solutions will not foam. Circular G.D.C. 162.

• CYANAMID BOOKLET

American Cyanamid & Chemical Corp., 30 Rockefeller Plaza, New York, N. Y., has recently issued a booklet entitled "Aero Brand Tetra Sodium Pyrophosphate." The material described therein is said to combine a number of unique properties which enable it to be used in a wide variety of products, for example: soap, detergents, clay refining, dairy cleaning, textiles and asphalt emulsions. Its textile uses include the following: *silk*—in the degumming of silk Aero brand tetra sodium pyrophosphate can be used to replace a portion of the soap in removing sericin gum from the raw silk; *rayon*—the material is used in scouring of woven and knit goods in conjunction with soap; *cotton*—the material is used in the kier boiling of grey goods and in the scouring of woven or knit goods; *wool bleaching*—used as a stabilizer of peroxide bleach baths. Copies of the booklet are available upon request.

• JOIN IMPREGNOLE

Chas. M. Hayman, general manager of the Impregnole Corp., announces the appointment of Miss Nannene Gowdy and Edward Starks to the staff of the Impregnole Corp. In order to expand its services to the cutting-up and fabric trade, Miss Gowdy joined the Impregnole Corp. as technical advisor for its products "Weather-Sealed" by Impregnole, a water-repellent, and Per-San, a permanent fabric finish and also an antiseptic finish. Miss Gowdy has previously been head of the New York laboratories of Montgomery, Ward and more recently was director of the bureau of standards of Montgomery, Ward in Chicago. Mr. Stark will be one of the sales representatives in rain, sports, snow togs and street wear for Impregnole "Weather-Sealed" products. The offices of the corporation are at 580 Fifth Ave. and the plant is at West Warwick, R. I.

• GENERAL SALES MANAGER

E. W. Reese has been appointed general sales manager of Emery Industries, Inc., Cincinnati, Ohio. Mr. Reese has been affiliated with the manufacturing and sales departments of the Emery Industries for the past 16 years, and enjoys a wide acquaintance in the various trades served by Emery Industries. Mr. Reese succeeds K. L. Schanbacher who has resigned.

• TO WORK FOR WILLKIE

Edgar M. Queeny, president of Monsanto Chemical Company, announced recently that effective July 1st and until the November election, he will serve his company only half time and will receive only half pay, so that he can devote the balance of his time and energy in behalf of the presidential candidacy of Wendell L. Willkie.

Mr. Queeny revealed that at the last meeting of the Monsanto board of directors on June 20 he requested that he be granted a full leave of absence without pay and that upon refusal of the board to grant his request a compromise was reached whereby he will serve half time at half pay.

• ATLAS OMETER GOLF TROPHY

With a low gross score of 78 in a field of 91, Elmer F. Smith, manager of the Chicago office of American Aniline Products, Inc., won the Atlas Ometer Trophy at the annual outing of the Mid-West Section of the A.A.T.C.C., Lake Lawn, Delavan, Wisconsin, June 22. The cup, donated by Atlas Electric Devices Company, will remain in Mr. Smith's possession until next year when it will again be available for further competition. It will become the permanent property of the member who wins it three times. A new cup will be presented by Atlas when that time occurs.

• KELCO PROMOTION

W. R. Kiely, sales manager of Kelco Company, manufacturers of Algin products, announces the promotion of J. C. Reisinger who formerly covered the southern territory to manager of the Algin Division of Kelco Company with headquarters in New York. Mr. Reisinger will devote his time to the introduction and promotion of new Kelco Algin products. Mr. Kiely also announced the addition of T. C. Roberts, Jr., to the Kelco sales force to cover Mr. Reisinger's former territory in Maryland, Virginia, and North Carolina.

• COTTON TECHNOLOGISTS SOUGHT

The United States Civil Service Commission has announced examinations to secure cotton technologists for employment in the Bureau of Agricultural Chemistry and Engineering, and Bureau of Home Economics of the Department of Agriculture. The examinations cover four grades, with salaries ranging from $2,600 to $4,600 a year, less a retirement deduction of 3½ per cent.

Applications must be filed with the Commission's Washington office not later than August 12 if received from States east of Colorado and not later than August 15, 1940, if received from Colorado and States westward.

Applicants must have completed a 4-year course in a textile school or in a college or university of recognized standing with major study in textile technology, textile or mechanical engineering, chemistry, or physics. In addition they must have had responsible experience in cotton technology, part of which must have been in one of the following optional branches: Yarn and fabric manufacturing; textile chemistry, dyeing and finishing; fiber technology. For some of the grades and optional branches substitution of additional experience in cotton technology may be made for part of the education; and appropriate graduate study may be substituted for part of the experience.

Further information regarding the examinations, and application forms, may be obtained from the Secretary of the Board of U. S. Civil Service Examiners at any

first- or second-class post office, or from the U. S. Civil Service Commission at Washington, D. C.

• NEW SOUND MOVIE

How chemical coatings are vital considerations in modern industry and how they are important to everyday living is explained in the new all-color, sound movie, "More Than Meets the Eye," released this month by Interchemical Corp.

Chemical coatings—inks, industrial finishes and enamels, textile colors, and other protective and decorative materials—are shown in this film to be important sales influences. It is pointed out that performance today is often taken for granted in a product. But the appearance of the article can "make or break" a sale, whether the product be a refrigerator, a household appliance, a magazine, a printed shirt, or a dress fabric.

In many cases, the chemical coating may be important as adding to the life of the product. For example, when a woman buys a dress her first thought is usually "How does it look?" Then if she likes the pattern, she will want to know whether the colors will be fast to sunlight and whether they will remain bright after repeated washings.

"More Than Meets the Eye" describes the processes and research back of the manufacture of these coating materials and shows how the requirements of the ultimate consumer must be taken into account in the manufacturing operations. The enamel finish for a washing machine, for example, must produce a surface so tough that it will resist the chipping and marring and the various deteriorating forces to which the product will be subjected in the user's laundry.

The functions of the various divisions and subsidiaries of Interchemical are outlined in the picture. These units are engaged in producing printing inks, industrial finishes, textile colors, dry colors and pigments, press equipment, and various related products.

"More Than Meets the Eye" was produced by Willard Pictures, Inc., under the direction of George Welp, Interchemical Corporation.

The film has been prepared so that it will be of interest to manufacturers of products requiring chemical coatings and to the general public which uses these products. Since much of the picture is concerned with the production and application of industrial colors like printing inks, pigments, finishes, and textile colors, there are many spectacular color effects.

This movie is available for showings to manufacturers, associations, or groups interested in chemical coatings and their importance both in industry and in our daily living.

• SALES OF ZELAN

Sales of "Zelan" durable repellent finish have tripled within the past six months, E. I. du Pont de Nemours & Company announces.

Rainwear and cotton sports jackets, among the first garments treated with "Zelan," still take a big part of the production of the finish. Applications to cotton ski garments, golf jackets, hunting coats, fishing suits, raincoats and gabardine sides of reversible coats are increasing.

Among new uses for "Zelan" are organdie aprons, which are said to retain repellency to water and non-greasy stains after washing.

Umbrellas now treated with the material open an important field since they can be designed with many new types of fabrics. The development has enabled manufacturers to supply umbrellas to match women's accessories such as handbags.

Use of "Zelan" on sail cloth is increasing. Results of tests conducted last year with sails treated with "Zelan" which did not stiffen or bulk the fabric have created an active demand.

Yachting costumes of wind-resistant poplins, corduroys and spun rayons, to protect against spray, rain and fog are becoming an important outlet for the repellent.

Entrance of the finish into beach and play clothes open a promising field. Since volume of sales of these garments is large and increasing rapidly, it is felt that it soon will be a substantial part of total water repellent sales. The fresh appearance of all the usual types of these garments, spun rayon, cottons and poplins, is prolonged by reducing the absorption of moisture and perspiration.

Department stores in many sections of the country have used "Zelan" as a merchandising aid to increase sales of many types of clothes. Consumer interest is said to have been aroused by the following points: children's clothes are less likely to soil, and clean more easily; swimming trunks dry more quickly; hats, slack suits and dresses are less likely to stain; sports clothes treated with the repellent offer protection, without resorting to out-and-out rainwear. Retailers report demonstrations and promotion plans based on the water repellent enable them to sell high-grade garments quickly.

• TECHNICAL NOTES FROM FOREIGN SOURCES •

Structure of Cellulose and Other Polymers Related to Simple Sugars

W. N. Haworth—J. Soc. Dyers and Col., 56-49, *February*, 1940.—This is the Jubilee Lecture of the Society of Chemical Industry, delivered by Professor Haworth at Bristol in 1938, and reprinted here by permission (cf. Chem. and Ind., 1939, 58-917). The lecture discusses the manner in which sugar residues, the building units, are linked together to form the complex molecules of such polysaccharides as cellulose, starch, glycogen, inulin and lichenni.

The distinguished author reviews in summarized form the work of the past twenty years on the finer structure of the simple sugars and their mode of linking with one another. Such an article cannot well be further condensed, and is mentioned here as a convenient reference to the presentation of this complex subject by an outstanding authority. The text is illustrated by a number of diagrams and structural formulas, including several extremely interesting "skeleton models" of glucose, maltose, cellobiose, etc., both as single and as repeating units.

The Modification of Cellulose by Oxidation

G. F. Davidson—J. Soc. Dyers and Col., 56-58, *February*, 1940.—This paper was originally prepared for presentation at the Symposium on the Chemistry of Cellulose, a contribution from the British Cotton Industry Research Association. Its object, as stated by the author, is not to present an exhaustive summary of the extensive literature dealing with the action of oxidizing agents on cellulose, but to review some of the recent work in this field.

The term "oxycellulose" may be considered as referring to a cellulosic material whose chemical and physical properties have been modified by oxidation; the insoluble products of the hydrolytic action of acids on cellulose are referred to as "hydrocellulose."

One of the most important changes in the chemical properties of cellulose that results from oxidation is the development of reducing power towards alkaline copper solutions. This reducing power is expressed as the "copper number," which is defined as the weight in grams of copper reduced from the cupric to the cuprous state by

100 grams of the cellulosic material. The reducing power of oxycellulose is usually attributed to the presence of aldehyde groups, but as the measurement is not capable of stoichiometric interpretation, it is not possible to infer the content of aldehyde groups from the copper number.

When an oxycellulose is boiled with dilute alkali, its copper number is diminished, and at the same time it suffers a loss of weight which is approximately proportional to the copper number. As the proportionality factor is the same for all oxycelluloses, irrespective of the method of oxidation, the loss of weight on alkali-boiling has thus the same analytical significance as the copper number.

The oxidation of cellulose frequently results in an enhanced affinity for basic dyes, and the measurement of the preferential absorption of the basic dye Methylene Blue from solutions of its hydrochloride has played an important part in the study of oxycellulose. A high Methylene Blue absorption on the part of oxycellulose is accompanied by a strong tendency to retain alkalis. Such absorption of Methylene Blue by an oxycellulose is generally ascribed to the presence of acidic, presumably carboxyl, groups, but its measurement, like that of the copper number, cannot be interpreted stoichiometrically. Oxycellulose characterized by an enhanced absorption of basic dyes also exhibits a diminished affinity for direct dyes when compared with an unmodified cellulose.

Two extreme types of oxycellulose have been recognized, the reducing and the acidic, between which all possible gradations may be observed. Those of the extreme reducing type are characterized by a high copper number and excessive loss of weight on boiling with alkalis, but their Methylene Blue absorption is no greater than that of the original, unoxidized cellulose. The acidic type, on the other hand, exhibits a great affinity for Methylene Blue and abnormal retentive powers for alkali, accompanied by a low copper number. As a rough generalization, it may be said that oxidation in an acid medium gives rise to oxycelluloses in which the reducing character predominates, whereas oxidation in presence of alkali leads to the acidic type of oxycellulose.

Examples of oxycellulose formation have been studied in which the reaction between cellulose and an oxidizing agent is greatly accelerated by the simultaneous oxidation of a third substance present in the system. For instance, if cellulose is treated with a solution containing potassium dichromate and oxalic acid, both the cellulose and the oxalic acid are oxidized, and the oxidation takes place much more rapidly than it does with bichromate in the presence of sulfuric acid. This method provides one of the most convenient ways of preparing an oxycellulose of the extreme reducing type.

Oxycellulose formation is always accompanied by a fall in the tensile strength of the fiber, and in some cases by an additional loss of strength when the oxidized material is submitted to an alkaline treatment. As a guide to the loss of strength that an oxidized cellulose has suffered, the copper number is valueless unless the method of modification and the subsequent history of the material are known. A rise of one unit in the copper number may correspond to a negligibly small or practically complete loss of strength, according to whether the oxycellulose belongs to the reducing or to the acidic type.

The measurement of the viscosity of solutions of cellulosic materials in cuprammonium has been established as the most generally useful method of assessing the extent of modification resulting from the action of acids or oxidizing agents. In investigations made of the relation between the tensile strength and the viscosity of modified cellulose, it was found convenient to employ the reciprocal of the viscosity, i.e., the fluidity, to express the results of measurements of rates of flow of cuprammonium solutions, for it was shown that the relation between the fluidity of 0.5 per cent solutions of modified cottons and the percentage loss of strength was approximately linear.

The development of the molecular-chain theory of the structure of cellulose has made it possible to interpret theoretically much of the behavior of modified cellulose that was formerly obscure. The viscosity of synthetic or naturally occurring linear polymers is now believed to be a function of the average chain-length, so that increase of fluidity, as also loss of tensile strength, of a cellulose resulting from the action of acids or oxidizing agents indicates a diminution of the molecular chains. Since both fluidity in cuprammonium and tensile strength are functions of the chain-length, it might be expected that all methods of modification of cellulose would lead to the same relation between strength and fluidity. As this has been found not to be the case, it has been suggested that the oxidation of cellulose does not in general lead directly to rupture of the chain molecules, but renders the linkages adjacent to points of attack abnormally susceptible to hydrolysis, particularly by alkalis. Different effects are therefore observed in oxycellulose formed in acid or in alkaline medium. A study of the fluidity in acetone of cellulose nitrates prepared from various types of modified cotton has served to confirm this hypothesis of alkali-sensitive linkages.

In spite of the great amount of work which has been done on the modification of cellulose by oxidation, very little is known with regard to the chemical reactions underlying oxycellulose formation. Most of the hypothesis advanced have involved the assumption that the oxidative attack occurs at the primary alcohol groups in the cellulose chain molecules. This group, it is supposed, is oxidized first to an aldehyde group, and then, under favorable conditions, to a carboxyl group. If the oxidation is limited to the first of these reactions a reducing oxycellulose results, whereas the two consecutive reactions lead to the formation of an acidic oxycellulose. Recent investigations in the use of different oxidants, however, have suggested some possible modification of this view.

Considering the diversity of products which have been obtained by the action of the various oxidizing agents, it seems certain that the oxidation of cellulose is, in general, a complicated process, but it may well be that the ways in which the primary oxidative attack on the cellulose chain molecule can occur are confined to a few simple reactions.

Evaluation of
Special Finishes
for Textiles*

EPHRAIM FREEDMAN**

NATURAL textile fibers have done well for man since time immemorial. As man's culture developed the requirements which his textile fabrics had to satisfy became increasingly difficult.

During the early part of the 19th century it became apparent that a new branch of textile technology would have to be inaugurated. Either it would be necessary for man to create new fibers or he would have to modify his available textile materials so that their use could be extended to satisfy his ever increasing demands upon them.

In both fields, outstanding successes have been attained. Synthetic materials now vie with textiles made of cotton, wool and silk, while special finishes applied to natural as well as to synthetic textiles expand their usefulness and increase their value to man.

The subject of this talk concerns itself with the evaluation of some of these special textile finishes.

Textile finishes may be divided into three major classifications—

1. Those produced mechanically e.g. preshrinking.
2. Those which alter the chemical nature of the fiber, e.g. cotton mercerization or wool chlorination.
3. Those involving the deposition of substances in or on the fiber or the fabric, e.g. starch, synthetic resins or rubber.

The methods of application to the fiber are dependent upon the nature of the product and the result desired.

The specific purposes for which the special finishes are employed are numerous and varied. Finishes have been developed which made textiles antiseptic and odor-retardant while other finishes reodorize or impart masking odors to them. Some finishes when applied to textiles, prevent or lessen the degree of crushing, creasing, shrinking, rotting, water-penetration, or staining. Some finishes make textiles flame proof, or fire-retardant. Finishes are employed to inhibit the tendering action of sunlight, to prevent or retard gas-fading, to produce gloss and to dull fabrics, to produce crinkly and moire effects, to make textiles water-absorbent, to render them more resistant to abrasion, to running, snagging, or scuffing.

I will consider some of these and develop them in more detail but for obvious reasons will purposely refrain from mentioning any of them by brand name.

Antiseptic finishes usually consist of germicidal agents absorbed by or deposited on the textile. They may render the textile bacteriocidal or bacteriostatic, i.e., they may kill germs or merely inhibit their growth. Because of their germ-inhibiting properties they retard the decomposition of perspiration and therefore as long as they are present in sufficient amounts, prevent the formation of "body" odors.

Some of these antiseptic finishes are less soluble than others which means that they may not disclose a great germ-inhibiting effect on contacting materials. Nevertheless this particular property prolongs the useful life of the finish because it is not readily leeched out and dissipated.

The evaluation of antiseptic finishes is predicated therefore upon—

(a) their ability to remain germ free.
(b) their destructive action upon germs.
(c) the permanence of the finish.

Because germicidal agents are frequently highly toxic or capable of producing skin irritations, or of being absorbed by the skin and producing skin irritations, or of being absorbed by the skin and producing harmful effects, it becomes very important to subject antiseptic finishes to biological tests over a prolonged period of time before proceeding to market them.

The literature contains a number of references to the method for achieving crease-resistance of textiles. Values of crease angles at 65 per cent relative humidity for some of the textile fibers are reported as

Viscose rayon	60°- 65°
Cuprammonium rayon	65°- 70°
Acetate rayon	95°-105°
Silk	120°
Wool	120°
Cotton and linen	75°- 90°

Crush resistance of pile fabrics particularly velvets is very important. The ratio of the pressure required to compress the pile (C) and the energy of recovery (R) is used by one investigator to provide a compression resilience factor $= \frac{R}{C}$.

The permanence of crush resistant finishes should be taken into consideration in the evaluation of the merchandise; the effect of dry-cleaning and washing should be observed.

*Portion of a paper presented at meeting, American Association of Textile Technologists, June 5, 1940.

**Director, Macy's Bureau of Standards.

Treatments to prevent moth damage are numerous. Hundreds of different chemicals and many different processes for applying moth proofing agents are employed. Some of the moth proofing products offered in recent years have been toxic to man rather than to moths. Other moth proofing products have been of rather doubtful value. Investigators are still in disagreement as to the best methods to be employed for the evaluating of moth proofing agents. Some prefer to run tests in petrie dishes or glass jars with moth or carpet beetle larvae in immediate contact with the treated fabric. Others consider the use of placing both treated and untreated fabrics in the same vessel. Some prefer to use large cabinets wherein are hung specimens of the treated materials. At the bottom of these cabinets we find feathers abounding in moth life. The millers fly around in the cabinet and deposit some of their eggs on the materials, much as they would do under actual conditions of textile storage in the home. This test of course is probably not as severe as the petrie dish or jar tests. Some investigators prefer to attach primary importance to the amount of moth excreta collected after a pre-determined test period. Others prefer to place maximum emphasis upon the degree of damage done to the textiles. One investigator prefers opening the larvae and examining the alimentary contents. If the fabric has gone more than ½ the length of the canal, the toxicity of the moth proofing agent is considered too low.

The effect of washing and dry cleaning on the permanence of moth proofing agents requires serious consideration. Materials that are normally washed, should be treated with wash-resistant moth proofing agents, while those that will normally be dry cleaned should be given special treatment to withstand the cleaning operation.

Certain fibers become tender upon exposure to light. To prevent this action certain manganese salts have been employed. Also phenolic and ureic resins and other products have been used. The evaluation of the light resistance of these materials would include Fade-ometer tests on washed and dry cleaned samples as well as on the original fabrics.

Water-resistant finishes for textiles have been on the market for many years. Until recently these finishes had been removed by dry cleaning and some of them had been removed by washing.

Recently there have appeared on the market water-resistant finishes which resist a number of dry cleanings and washings thereby lengthening the serviceability of the article.

Numerous textiles such as shower curtains, awning material, glider covers and tent materials are often subjected to long contact with water under subdued light conditions thereby encouraging the growth of mildew and so resulting in the rotting and tendering of the fabric.

The application of mildew and rot-retardant finishes has helped materially to increase the serviceability and therefore the sale of this type of finish.

For years many curtain materials, drapes and furniture covers have been treated with water soluble finishes to produce glossy and crisp effects, which unfortunately were removable by water. The so-called permanent finishes have in a number of instances superseded these old type finishes by virtue of the fact that they impart to texile fabrics a gloss and crispness capable of withstanding innumerable washings.

Any number of products have appeared with accompanying claims to the effect that they render textiles more resistant to abrasion; in the case of stockings to running and snagging and in the case of pyroxylin coated materials to scuffing, creasing and folding. The value of these finishes varies considerably.

Recently, we have seen an increase in the number of brushed rayon garments and blankets. These materials frequently represent a fire hazard because they encourage the rapid spread of flame.

Numerous products have been developed to make them flame retardant. The problem confronting the producers of these finishes and those that apply them, requires that they take into consideration the hand and the appearance of the fabric and the harmlessness of the flame retardent agent. The additional problem confronting the retailer is the permanency of the finish. If the finish is removed by washing or dry-cleaning, then of course the article will, upon being washed or dry-cleaned lose their property and again present a fire hazard.

The creation of a permanent flame-retardent finish for textiles will not only increase the sale of brushed rayons but will also eliminate the fire hazard accompanying the use of cotton blankets, numerous curtains and other drapery materials and sheer rayon and cotton garments.

I have just given you a brief outline of the scope of special finishes and some of the angles pointing to their evaluation.

During the progress of my discourse, I refrained, with a few exceptions, from discussing the consumer approach to these special finishes.

I shall now discuss this approach with the object of encouraging the use of a similar approach to the problem of special finishes and their application to textiles by those who manufacture the finishes and by those who apply them.

Needless to say, consumers have benefited materially because of the creation and application of special finishes for textiles. Parenthetically speaking, manufacturers and retailers have also benefited greatly because the application of these special finishes has increased the range of usefulness of textiles and therefore has resulted in marked increase of sales.

However, there is much more to this problem than just making sales.

If the textile industry is to retain the good will of consumers, it behooves the industry to engage first in research which will disclose whether the finish is capable of producing the effects or of offering the protection for which it has been expressly created. Secondly, the application

of each finish should be restricted to the type of textile fiber to which it imparts a substantial effect. Thirdly, the finish should be subjected to biological tests to make certain it will not prove injurious under conditions of use. In this connection it is well to remember that salts of arsenic, mercury and lead are likely to prove harmful. Fourthly, the permanence of the finishes under conditions incidental to use should be studied and that degree of permanence should be clearly set forth on labels or tags affixed to the merchandise. If the finish deteriorates with time or is removed by washing or dry-cleaning, the label or tag should carry that information. If ironing affects the value of the finish that should be disclosed.

Regardless of the kind or quality of finish, the terminology used to describe it should be simple and factual. The term "proof" applied to materials treated to resist moth damage is, to say the least, an exaggeration of the truth. The term "repellant" to describe a material treated to resist the passage of water is a misnomer. Bats, racquets, or clubs are used to repel oncoming balls. Shower curtains, raincoats, umbrellas, and glider covers do not repel water. If properly treated, they resist the passage of water. An article may be fire retardant because it does not encourage the spread of flames or sparks, but that retardant property does not necessarily make it "fire proof" because it may itself be consumed by fire.

Just because a finish retards or inhibits the growth of bacteria, it does not warrant the use of the term "antiseptic" without further qualifying descriptive terminology because the term "antiseptic" when used alone implies the ability to kill off all germ life. The term "deodorize" should not be used to describe a product which masks one odor with another. "Reodorize" is the more accurate word.

The fact that an antiseptic finish inhibits the development of body odors or any other odors on textiles does not mean that it deodorizes. It merely means that it prevents the creation of these body odors.

If consumers are to be favorably disposed toward retailers, and if consumers and retailers are to be favorably disposed toward manufacturers, it would help if these attitudes which I have just enumerated were given increasing weight at the point of manufacture of the finish and also at the finishing plant. Many special finishes for textiles have a definite reason for existing. Their application to textiles can be increased and their usefulness can be broadened with greater effect if the consumer approach to the evaluation of these special finishes begins with the manufacturer.

Cation Exchange —

(Continued from page 360)

smooth curves in this figure are not drawn to fit these points, but are explained in Section III—6. It should be noted that the data obtained by the subtraction process described above are not subject to errors inherent in the method of correction for the selective sorption of water by the samples, since the subtraction would presumably cancel any such errors. Furthermore these results should not be susceptible to secondary effects (Section III—2) for the same reason. As a result, it is not surprising that a somewhat more definite maximum is obtained. The curve approaches a maximum value of 0.058 milliequivalent per gram.

3. The Hydrogen Ion Equivalence of the Cationic Ashes of Dewaxed and of Lime-washed, Depectinized Cottons

The hydrogen-ion equivalence of the cationic ashes of several different samples of cotton are given in Table 3. A comparison of the data for the ash determination on dewaxed cotton with those on dewaxed cotton which had been washed with lime water and then with distilled water indicates that all of the acidic groups in the former existed in the form of salts. The data also show that if the extraction is extended from 4 to 8 hours, the cationic ashes of the lime-washed samples remain unaltered. The difference between the mean values of the cationic ashes of dewaxed and of lime-washed, depectinized cottons is 0.054 milliequivalent per gram. This represents the cationic ash bound by the pectic substance.

A comparison is given in Table 4 of the maximum acid-

TABLE 3

The Hydrogen-ion Equivalence of the Ashes of Various Cotton Samples

Sample	*Cationic Ash* *Milliequiv./g.*
Dewaxed cotton	0.063
Lime-washed dewaxed cotton	.064
Lime-washed depectinized (4 hr.) cotton	.010
Lime-washed depectinized (8 hr.) cotton	.010

TABLE 4

Comparison of the Maximum Acid-binding Capacities of Various Samples with Their Contents of Cationic Ash

Sample	*Maximum Acid-binding Capacity* *Milliequiv./g.*	*Cationic Ash* *Milliequiv./g.*
Dewaxed cotton	0.066	0.063
Lime-washed depectinized cotton	.008	.010
Pectic substance (calculated)	.058	.054

binding capacities of dewaxed cotton, lime-washed, depectinized cotton, and the pectic substance with their respective cationic ashes. The correspondence is very close for each sample. In addition to the excellent agreement obtained by the two methods, these values also agree well with independent determinations.* Calculation of the data of Walker and Quell[2] for the total cationic ash as obtained by direct methods, in samples of water-washed cotton, gave a value of about 0.064 milliequivalent per gram. Similarly, direct determinations of the uronic acid-carboxyl content, by the method of Whistler, Martin and Harris[7] of the cotton used in this work gave a value of 0.055 milliequivalent per gram. Since the entire uronic acid-carboxyl content of the cotton can be accounted for by titratable groups, the results suggest that the pectic substance exists on the fiber as the salts of pectic acid.

4. The Temperature Coefficient of the Titration Curve of Dewaxed Cotton

Table 5 gives the data for the titration curve at 25° C.

TABLE 5
Combination of Dewaxed Cotton with Hydrochloric Acid at 25° C. in the Absence of Added Salt

pH	*Acid Bound Milliequiv./g.*
1.45	0.058
1.93	.058
2.45	.053
3.04	.041
3.75	.027
4.49	.012
5.32	.004
6.38	.001
6.54	.000

of dewaxed cotton in the absence of added salt. These data are compared with the corresponding data for dewaxed cotton at 0° C. in Figure 3. The results indicate that there is no measurable temperature coefficient of the

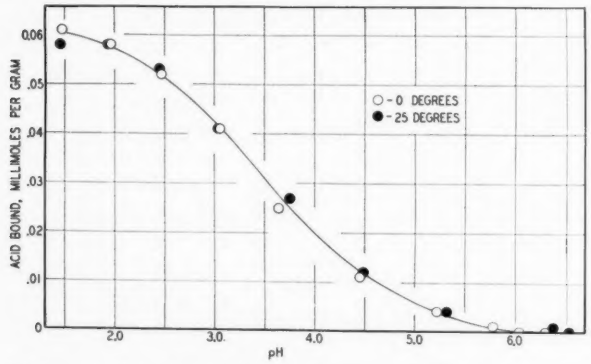


Figure 3. Titration curves of dewaxed cotton at 0° C. and 25° C.

equilibrium values in this range of temperature, a result in keeping with the identification of the acidic groups of the fiber as carboxyl groups[11].

5. Significance of the Acid Groups in Depectinized Cotton

The results in the foregoing sections show that about 85 per cent of the acidic groups in dewaxed cotton can be allocated to the pectic substance. The question arises, however, as to the source of the acidic groups in the depectinized cotton. The following explanations for their presence might be offered:

(1) Removal of the pectic substance by the alkali may be incomplete.
(2) The protein fraction of the cotton may have an appreciable acid-binding capacity.
(3) The acidic groups may be present in low molecular weight degradation products of cellulose.
(4) The acidic groups may be an integral part of the cellulose itself.

A comparison of the acid bound by samples of cotton which had been extracted with sodium hydroxide for 4 and 8 hours showed that both bound essentially the same maximum amount of acid. Since it has been shown by direct analyses for uronic acids that both samples were free of detectable amounts of pectic substance[12], the first of the three possibilities is immediately ruled out. Furthermore, since increasing the duration of the alkali-treatment lowered the nitrogen content from 0.06 to < 0.015 per cent, and the maximum acid-binding capacities of the two samples are the same, it is difficult to see how the protein fraction could account for any substantial portion of the acid bound. The third possibility also appears unlikely because it would be expected that acidic compounds of low molecular weight would be readily removed during the treatment with alkali.

It therefore appears that the residual acidic groups in depectinized cotton may well be an integral part of the cellulose molecule, as for example, an end-group. Further work, which is necessary for establishing the validity of such a conclusion, is now in progress in this laboratory. By making the tentative assumption that these acidic groups are contained in the cellulose molecule and are present to the extent of about 0.010 milliequivalent per gram, as determined experimentally, it is found that the cellulose has an equivalent weight of about 100,000, which corresponds to a minimum chain length of about 600 glucose residues.

6. The Application of the Donnan Theory to the Titration Curves of the Pectic Substance

As shown in Figures 1 and 2, addition of salt produced shifts in the positions of the titration curves to lower pH values. The direction of this shift and the fact that it is not measurably augmented by an increase of ionic strength to 1.0 *M* suggest that the results may be explained by the Donnan equilibrium. Calculation shows that any alternative mechanism involving the competition of cations for the carboxyl groups requires a still further shift with increased concentration of these ions.†

*Further investigations now in progress in this laboratory, indicate that these values are also approximately checked by direct titration with alkali, of samples of cotton freed of cationic ash by electrodialysis.

†The titration curves for wool have been shown to be consistent with an ion-association mechanism which involves the combination of cations other than hydrogen ions with basic groups, and anions other than hydroxyl ions with acidic groups in the wool[13]. Since, according to this view, ions of unlike sign are bound by the fiber, the differences in ion concentrations inside and outside the wool phase which would be predicted

The derivations of the Donnan equations for the titration curves in the absence of salt and in solutions of hydrochloric acid and potassium chloride of 0.1 *M* ionic strength are given below. Bracketed quantities are molar concentrations. The density of the phase which consists of the pectic substance is taken as unity. The subscripts "o" and "i" refer to quantities in the outside (solution) and inside (pectic substance) phases, respectively. R is the ratio of the activity coefficient of an ion and in the inside phase to its activity coefficient in the outside phase, referred to the same standard state, and is assumed to be the same for all the ions involved. This is a reasonable assumption only when all the ions bear similar charges. K is the ionization constant of the acid, α is the fraction of the total acid $[A^-] + [HA]$ in the ionized form $[A^-]$, V represents volume, and ρ equals $[H^+]_i/[H^+]_o$. The acid groups were assumed to be present as salts of the divalent metal M^{++}. The positions and shapes of the curves are not affected much by the valence of M, in any case. The equations follow:

In the absence of added salt:

$$3.\quad \frac{[H^+]_i R \alpha}{1-\alpha} = K$$

$$4.\quad [H^+]_o [Cl^-]_o = [H^+]_i [Cl^-]_i R^2$$

$$5.\quad [M^{++}]_o [Cl^-]_o^2 = [M^{++}]_i [Cl^-]_i^2 R^3$$

$$6.\quad 2[M^{++}]_o + [H^+]_o = [Cl^-]_o$$

$$7.\quad [A^-] + [Cl^-]_i = [H^+]_i + 2[M^{++}]_i$$

$$8.\quad 2\frac{V_o}{V_i}[M^{++}]_o + 2[M^{++}]_i = [A^-] + [HA] = 0.055$$

These reduce to

$$9.\quad \frac{\{[A^-] + [HA]\}\rho R^2\alpha^2 - \{[A^-] + [HA]\}(\rho^3R^3 - 1)\alpha}{\rho^2R + \frac{V_o}{V_i}} + \frac{(\rho^2R^2 - 1)K}{\rho R}(\alpha - 1) = 0$$

$$10.\quad pH = pK + \log\frac{\rho R \alpha}{1-\alpha}$$

In hydrochloric acid-potassium chloride solutions of 0.1 *M* ionic strength:

$$11.\quad \frac{[H^+]_i R \alpha}{(1-\alpha)} = K$$

$$12.\quad [H^+]_o [Cl^-]_o = [H^+]_i [Cl^-]_i R^2$$

$$13.\quad [M^{++}]_o [Cl^-]_o^2 = [M^{++}]_i [Cl^-]_i^2 R^3$$

$$14.\quad [K^+]_o [Cl^-]_o = [K^+]_i [Cl^-]_i R^2$$

$$15.\quad [K^+]_o + 2[M^{++}]_o + [H^+]_o = [Cl^-]_o = 0.1$$

$$16.\quad [A^-] + [Cl^-]_i = [H^+]_i + [K^+]_i + 2[M^{++}]_i$$

$$17.\quad 2\frac{V_o}{V_i}[M^{++}]_o + 2[M^{++}]_i = [A^-] + [HA] = 0.055$$

These reduce to the following equations, neglecting $2[M^{++}]_o$ in equation 15:

$$18.\quad \alpha = \frac{0.1(\rho^2R^2 - 1)}{\{[A^-] + [HA]\}\rho R^2} + \frac{\rho^2R}{\rho^2R + V_o}$$

$$19.\quad pH = pK + \log\frac{\rho R \alpha}{1-\alpha}$$

Figure 4 shows the calculated acid titration curves for

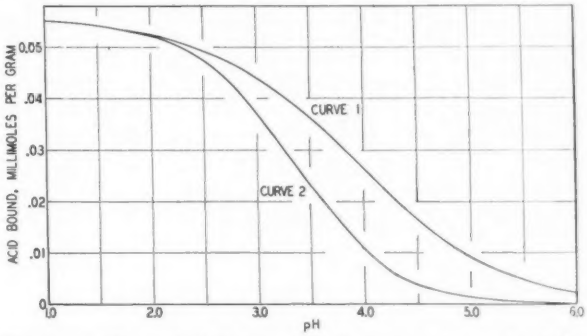


Figure 4. Theoretical Donnan titration curves for an insoluble acid of pK 3.3 in the absence of added salt,—curve 1, and in solutions of 0.1 *M* ionic strength,—curve 2. The assumptions and conditions used are described in the text (Section III—6).

the salt of a carboxylic acid of pK 3.3 present to the extent of 0.055 milliequivalent per ml.* These curves were calculated by varying the parameter ρ, and taking R as unity. Curve 1 was calculated from equations 9 and 10 and curve 2 from equations 18 and 19. The latter curve is only very slightly altered by increasing the ionic strength from 0.1 to 1.0 M. From the shape of the curves and the direction of the pH shift (Figure 4), it is seen that the titration data for the pectic substance (Figure 2) are qualitatively consistent with the Donnan equilibrium, but the shift produced by the addition of salt is considerably smaller in the experimental curves than in the theoretical. It is interesting to note that the experimental data (circles) of Figure 2 can be fitted very closely with calculated curves (solid lines) obtained by taking R equal to 0.05. It should be noted that R is present in each term of equation 9, 10, 18 and 19 in the relationship R/V_i. Therefore, the adjustment of either R or V_i can eliminate the discrepancy between the theoretical curves of Figure 4 and the experimental data of Figure 2. However, V_i has been fixed at the highest reasonable value by taking a value of unity for the specific volume, and the rather improbable value of R equals 0.05 is therefore required to make the calculated curves conform with the data.

Aside from the a priori unlikelihood of this value of R the agreement of the calculated curves with the data is subject to question on the following grounds. The solid lines of Figures 2 and 4 represent the calculated amounts

by the simple application of the Donnan equations (taking into account the binding of hydrogen ions alone) would tend to be minimized, and may apparently be neglected. With cellulose, which is not amphoteric, there is no clear evidence for the existence of association of cations with carboxylate groups and a Donnan effect is therefore to be expected.

*The value of 0.055 milliequivalent per gram is the mean of the values for the maximum acid-binding capacity, cationic ash, and uronic acid carboxyl content of the pectic substance (section III-3).

of acid fixed by the fiber by exchange with cations held by carboxyl groups. Experimentally determined acid-binding capacities are a measure of the *total* amount of acid adsorbed by the solid phase. If the value for R were 0.05, it would have as a consequence the presence within the solid phase of large quantities of dissolved acid at low values of pH, with resulting large discrepancies between the calculated and experimental values for the total amount of acid adsorbed. If the pectic substance is thought of as being dispersed throughout the fiber, which is rather unlikely[14], the circles of Figure 2 would represent only the acid fixed by the carboxyl groups, since they were calculated by a subtraction process that should cancel the effect of any acid dissolved in the fiber. For such a case, however, calculations show that there would be a sharp rise, not found by experiment, in the total amount of acid adsorbed by both the dewaxed and depectinized cotton at low values of pH (Figure 1). If, on the other hand, the pectic substance is thought of as existing in a shell around the fiber, the subtraction process should not cancel the effect of the acid dissolved in the shell, and the circles of Figure 2 should show a sharp rise at low values of pH, which was also not found experimentally. The quantitative agreement of the data of Figure 2 with the curves cannot therefore be accepted as being too significant, but it is clearly shown that they are consistent with the Donnan equilibrium.

Because of the form of the function, the equation for the titration curve in the presence of salt (curve 2, Figure 2) is virtually identical with the equation for the mass-action law for a monobasic acid of pK 3.3. This is merely an algebraic statement of the fact that the Donnan treatment predicts that as the concentration of salt is increased, the differences between the ion concentrations of the inside and outside phases disappear, and a simple mass-action law is approximated. It is reasonable that the data for pectic substance in the presence of salt should be thus fitted by the mass-action law for an acid of pK 3.3, since pK values for other polyhydroxy acids are near this figure. Thus, glyceric and gluconic acids, e.g., have pK values near 3.5[15].

The authors are indebted to John Beek, Jr., for assistance in formulating the equations for the Donnan equilibrium.

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